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THE EFFECT OF DIFFERENT SURFACE TREATMENTS AND BONDING AGENT COMBINATIONS ON THE SHEAR BOND STRENGTH OF TITANIUM ALLOY TO LITHIUM DISILICATE GLASS-CERAMIC

MASTER THESIS

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ABBREVIATION AND SYMBOLS

Ti: Titanium alloy

CAD-CAM: Computer-aided design and computer-aided manufacturing

CP Ti: Commercial Pure Titanium.

mm: Milimeter

mm^2: Square millimeter

APA: Airborne Particle Abrasion

HF: Hydrofluoric acid

Psi: Pounds per Square Inch.

SBS: Shear bond strength

LED: Light Emitting Diode

SBS: Shear bond strength

N: Newton unit of force

MPa: Mega pascal.

ISO: International Organization for Standardization.

µm: Micrometer unite.

ANOVA: Analysis of variance.

Tukey HSD: Tukey method, honest significant difference test.

P: P Value (Calculated Probability).

MDP: 10-methacryloxydecyl dihydrogen phosphate.

et al: and others.

ABSTRACT

Statement of problem. The esthetic challenges of using titanium alloy abutments have led to the increasing use of prefabricated titanium inserts bonded to tooth colored abutments and fixed dental prostheses. Optimal bonding protocol related to surface treatment of titanium alloy have not been established.

Purpose. The purpose of this in vitro study was to assess the effects of different surface treatment and cementation procedure combinations of titanium alloy disks on the bond strength of lithium disilicate glass-ceramics.

Material and methods. A total of 40 computer-aided designed and computer-aided manufacturing (CAD-CAM) titanium alloy disks (4×6.6 mm) were used. Specimens were divided into two groups (2 groups; n=20/ group) according to surface treatment type including alumina airborne- particle abrasion (50µm, 0.4 Mpa for 10 seconds at 20-mm distance) and 9.5% Hydrofluoric acid etching gel for 90 seconds. Heat-pressed lithium disilicate disks were fabricated and bonded with resin cements onto the treated surfaces after dividing each group into two subgroups (2 subgruops; n=10/subgroup) depending on the type of resin cement was used Multilink Hybrid abutment cement or Panavia SA plus cement . All the specimens were stored in the distilled water at 37℃ for 24 hours then 5000 cycles of thermal cycling were done. After thermcycling shear bond strength test was conducted using a universal testing machine. Statistical analysis were done by ANOVA and Tukey honset sinificant differences tests ($P < 0.05$).

Results. The mean of ±SD shear bond strength values ranged from 3.89±0.59 MPa to15.91±3.23 MPa.The highest shear bond strength values were obtained using airborne-particle abrasion. Resin cements tested in this study had similar effect on the bonding strength values.

Conclusions. Surface treatment of titanium alloy with sandblasting improved shear bond strength but increasing the etching time of 9.5% Hydrofluoric acid did not. The cement type didn’t have a significant influence on shear bond strength.

Key words: Titanium alloys, dental implant abutment, shear bond strength

1. INTRODUCTION

Titanium-6aluminum-4vanadium alloy (Ti6A14V) considered as a well accepted material for use in both temporary and permenant implant abutments because of its physical and mechanical properties, biocompatability, relatively low cost and suitability for computer-aided design and computer-aided manufacturing (CAD-CAM) (1). However, the metalic color of the alloy presents an esthetic problem, especially in submucosal peri-implant tissues (2). Therefore, zirconia abutments have been introduced to provide improved periimplant tisssue esthetics but also have been reported to fracture at the abutment neck and cause wear at the implant interface (3). In order to solve this problem and to achieve more natural esthetics, zirconia combined with titanium platforms have been introduced as bi-component abutments (4,5). In addition to that, recently Ti based abutments have been used in combination with different esthetic materials, including heat pressed lithium disilicate glass ceramic to give more translucent restorations (6,7). These hybrid abutments require cementation processes to bond the Ti -based material to the ceramic component.

Therefore, the bonding between the two materials considered as the crusial factor for the durability of the definitive prothesis. As Ti- surface is amenable to modification treatments such as airbrone- particle abrasion (8), and etching with hydrofloroic acid (9) that improve surface bond strength at Ti / resin cement interface. A clear protocol of Titanium surface treatment and cementation procedure are essential to maximize bond strength and the longevity of the permenant prothesis of the two diferent materials.

The aim of this in-vitro study is to assess the shear bond strength of lithium dislicate ceramics to Ti CAD/CAM disks, using two different surface treatments in combination with two types of cements. The null hypothesis of this study is that different surface treatment and cementation procedure combinations will not affect the shear bond strength values between the titanium alloy and lithium disilicate ceramic interfaces.

2. LITERATURE REVIEW

The ideal goal of modern dentistry is to restore the patient to normal contour, function, comfort, esthetics, speech, and health. A dentist provides this restoration for a living, whether removing caries from a tooth or replacing several teeth (10).

2.1. Prosthodontic dentistry

The branch of dentistry which deals with the replacement of missing teeth and related mouth or jaw structures by crowns, bridges, dentures, implant or other artificial devices Prostodontic has 4 main branches (10).

- Removable prostodontics
- Fixed prostodontics
- Maxillofacial prostodontics
- İmplant prostodontics

2.1.1. Removable prostodontics

The branch of prosthodontic that concerned with replacing the teeth and soft tissues with a non-permanent prosthesis that can be removed. These are often known as dentures, and can replace a full arch of teeth (complete dentures), or number of individual or grouped tooth spaces (partial dentures). The dentures are held in place using number of elements - complete dentures are held in place by forming a seal against the palate, and saliva can help significantly with this process. Partial dentures can be held in place by design (i.e. they are made to sit into areas which prevent it from falling out), or they can have a metal base, with little clasps which sit against the teeth and prevent dislodging. Sometimes the remaining parts of teeth can be used to help retain the denture, such as little magnets placed into the root surfaces of worn teeth. In all cases, the experience of the patient plays a big part in how the teeth are managed (10).

2.1.2. Fixed prostodontontics

It is concerned with restoring teeth using restorations that are fixed into the patient's mouth. They are typically made in a laboratory after taking impressions for the technician to work with. They are also known as 'indirect restorations'. These indirect restorations can be used to restore and repair single or multiple teeth, and can be used to restore relatively small spaces between the teeth. The main types of indirect restoration are crown, bridge, inlay, inlay, veneer (11).

2.1.3. Maxillofacial prosthodontics

It is dealed with the prosthodontic management of patients who present with congenital or acquired problems because of surgical intervention, trauma, pathology in the maxillofacial region. A wide variety of maxillofacial prostheses are being fabricated in practice. Based on the location, use, and area of restoration, maxillofacial prostheses can be classified as follows: intraoral prostheses which can be in the mandible or in the maxilla and extraoral prostheses (10).

2.1.4. Implant prosthodontics

The phase of prosthodontics concerning the replacement of missing teeth and/or associated structures by restorations that are attached to dental implants. An implant can be defined as, “A graft or insert set firmly or deeply into or onto the alveolar process that may be prepared for its insertion”. A dental implant is defined as, “A substance that is placed into the jaw to support a crown or fixed or removable denture (12).

It is indicated for completely edentulous patients with advanced residual ridge resorption, where it is difficult to obtain adequate retention, for partially edentulous arches where removable partial dentures may weaken the abutment teeth and also provide reduced masticatory efficiency, for single tooth replacements where fixed partial dentures can not be placed and when Patient's desire. Evidence of use of implants is seen in ancient civilisations like the Incas and Mayans. However, modern implantology has evolved from 1980 onwards. With the evolution of new materials for implants, implantology has come into widespread use. In 1940's, Dahlse introduced sub-periosteal implants. Later Linkow introduced the blade implants. In 1980's, it was Per Ingvar Branemark who introduced the concept of osseo-integration, which led to the widespread use of endosteal implants (12).

The increased need and use of implant-related treatments result from the combined effect of number of factors, including the following: 1. An aging population living longer 2. Tooth loss related to age 3. Consequences of fixed prosthesis failure 4. Anatomical consequences of edentulism 5. Poor performance of removable prostheses 6. Consequences of removable partial dentures 7. Psychological aspects of tooth loss and needs of aging baby boomers 8. Predictable long-term results of implant-supported prostheses 9. Advantages of implant-supported prostheses (13).

Today, a better teeth replacement option exists - the dental implants treatment because of their esthetics, comfort, stability, preservation of the adjacent teeth and the dental implants procedure can stop bone loss and improve the strength and functionality of your bite (14). As any treatment, the dental implant have some limitations including Patient affordability is the primary concern in the use of implants that cannot be used in medically compromised patients who cannot undergo surgery. Many patients do not accept longer duration of treatment and tedious fabrication procedures.It requires a lot of patient cooperation because repeated recall visits for after care is essential and it can not be universally placed due to the presence of anatomical limitations. Based on the materials used, the implants can be classified into Metallic implants (Titanium, Titanium alloy, Cobalt Chromium Molybdenum alloy) and Non-metallic implants (Ceramics, Carbon) (12).

2.1.4.1. Dental implant components

Dental implant parts include:

- Implant Body or fixture: is the component that is placed within the bone during first stage of surgery. It could be threaded or non-threaded. Threaded implant bodies are available in commercially pure (CP) titanium or as titanium alloys. The Ti or Ti alloys may be with or without a hydroxyapatite coating.
- Healing screw: During the healing phase, this screw is normally placed in the superior surface of the body. The functions of this component are to facilitate the suturing of soft tissue and to Prevent the growth of the tissue over the edge of the implant.
- Healing caps or healing abutments: Healing caps are dome-shaped screws placed over the sealing screw after the second stage of surgery and before insertion of the prosthesis.
- Abutments: Abutment is the part of the implant, which resembles a prepared tooth, and is designed to be screwed into the implant body. It is the primary component, which provides retention to the prosthesis. Titanium, precious alloys, alumina, and zirconia are availble for the fabrication of dental implant abutments (12). Historically implant abutments were manufactured in metal. The use of titanium abutments prevents the occurrence of galvanic and corrosive reactions in the implant/abutment interface, which enhances the peri-implant soft tissue health due also to its

high biocompatibility. However, excessive oxidation of titanium at ceramic melting temperatures and the low adhesion of the oxides to the surface of this material may be a problem in the titanium/porcelain systems. Metal abutments only solve partially the esthetical, functional and hygienic questions fundamental to the restorations over implants success (15). Three main categories of implant abutments are described, according to the method by which the prosthesis or superstructure is retained to the abutment: (1) an abutment for screw retention uses a screw to retain the prosthesis or superstructure, (2) an abutment for cement retention uses cemendental cement to retain the prosthesis or superstructure, (3) an abutment for attachement uses attachement device to retain a removable prosthesis such as an O-ring attachement (16) . In the past, two main types of abutments were available for restoring implants: pre-fabricated abutments, traditionally supplied by dental implant manufactures to match their respective implant systems, and custom abutments.

Recently, novel CAD/CAM abutments were introduced. CAD/CAM abutments can be custom designed to recreate the desired emergence profile and supporting crown orientation, facilitating the formation of anatomical mucosal topography and coronal contoures for prosthetic replacement. Specific computer software and milling machines utilize scan datd from he patient’s dental casts to fabricate a computer- generated abutments, milled from a block of titanium or zirconia. In general, all metal abutments have been reported to cause a greyish discoloration of the surrounding soft tissues, compromising the esthetic outcome in the anterior arches (17, 18). This discoloration is most apparent in patients with a thin gingival biotype that is incapable of blocking reflective light from the metallic abutment surface. Thus, ceramic abutments have been promoted to achieve better mucogingival esthetics (19). The first ceramic abutmemt “ceramic core” was introduced in 1993 in small and large dimeters (not commercially available) (20, 21). The abutm ent was a prototype of alumina ceramic with resistance to shearing forces that reached values up to those of the metal- ceramic crowns (22). Compared to metal abutments, these new abutments offered optically favorable characteristics, low corrosion potential, high biocompatibility, and low thermal conductivity (20). On the other hand, restoration made out of such ceramic cores were weaker when compared to metal-ceramic restorations (23).

Although a minimal strength value for ceramic abutments has not been defined, clinical studies have demonstrated sufficient fracture resistance of zirconia abutments in oral cavity for single-tooth replacement in anterior and premolar regions (24). Such controversies led to further investigations into new designs and materials for ceramic abutments. Further design improvements led to the application of a concept in which metals were used to reinforce the ceramic abutment and called hybrid abutment or bi-component abutment (25).This design was intended to provide an implant abutment that presented a metal reinforcements at the implant-abutment interface and thud provided improved aesthetics combined with increased resistance to fracture (25). Nowadays, pressed lithium dissilcate ceramics introduced as Hybrid abutment material which is luted to a Ti base. The shape, emergence profile and esthetic properties of such abutments can be ideally adjusted to the clinical situation.

- Impression posts: It is a small stem that facilitates the transfer of the intra oral location (of the implant or abutment) to a similar position on the cast. They are placed over the implant body during impression making laboratory Analogues.
- Prosthesis retaining screws: Prosthesis retaining screw penetrates the fixed restoration and secures it to the abutment.

- Implant super-structures: A super structure is the prosthetic component fabricated over the implant after its placement. At this stage, the implant that supports the prosthesis is considered as an abutment. Commonly used super structures include overdentures, fixed bridges, fixed detachable bridges and single crowns. Most super structures are connected to the implant via an attachment (10).

2.1.4.2. Titanium versus ceramics as dental implant abutments

Until today, metal implant abutments made out of titanium have been considered to be the ‘conditio sine qua non’ for the longevity of implant-borne reconstructions in all regions of the jaws. Clinical studies demonstrated excellent survival rates for fixed implant reconstructions supported by titanium abutments (26). Furthermore, in a recent systematic review, only a few complications were associated with metal abutments supporting fixed implant reconstructions. For this type of abutment, the most frequently occurring retrievable technical problem was loosening of the abutment screw (27). Nowadays, the esthetic outcome has become an additional criterion for the clinical success of an implant-borne reconstruction. One major drawback of metal abutments is their dark gray color. Several studies demonstrated a grayish discoloration of the peri-implant mucosa induced by metal abutments (28 ,29). Hence, although very stable from a technical point of view, metal abutments have limited indications in esthetically delicate areas (30). As an alternative, ceramic abutments made out of the high-strength ceramics alumina and zirconia were developed (31). Ceramic abutments offer several clinical advantages over metal abutments. Firstly, their esthetic benefit is well documented (32). Ceramic abutments induced significantly less mucosal discoloration than metal abutments (33). Secondly, less bacterial adhesion was found on ceramics such as zirconia than on titanium (34). Finally, the soft tissue integration of the ceramics alumina and zirconia is similar to that of titanium (35).

One shortcoming of ceramics is their mechanical behavior, as they are brittle and therefore, less resistant toward tensile forces. Micro-structural defects within the material may cause cracks in combination with tensile forces (36). An increase in the fracture toughness of a ceramic slows down crack propagation and consequently has a major influence on the material's long-term clinical stability (37). High-strength ceramics like alumina and zirconia exhibit very high fracture toughness,with zirconia exhibiting the highest fracture toughness of ceramics suitable for constructing abutments (38). To date, the reported clinical performance of alumina and zirconia implant abutments has been very promising. Alumina abutments supporting single crowns exhibited a 93–100% survival rate in anterior and premolar regions (39). Zirconia abutments supporting anterior and premolar single crowns even survived in 100% of cases in several studies (40). Furthermore, one recent randomized-controlled clinical trial (RCT) of zirconia and titanium abutments supporting single crowns in posterior regions reported a 100% survival rate for the ceramic abutments after 3 years (41). To date, facture of a zirconia abutment has not been reported in any clinical studies. Interestingly, loosening of the abutment screw was one of the few technical complications occurring at zirconia abutments (42). This finding is similar to the observations made at metal abutments. The mechanical strength of abutments made out of this ceramic seems to be adequate for clinical use as an alternative to metal abutments. To be suitable for clinical use as an alternative to metal abutments, ceramic abutments need to exhibit similar performance after a mean follow-up of at least 5 years (43).

2.2. Titanium and titanium alloy

The use of commercially pure titanium (CP Ti) and titanium alloys for dental applications has increased significantly since a description of its applications was first reported in 1977. These metals can be used for all-metal and metal-ceramic prostheses as well as for implants and removable partial denture frameworks. Titanium derives its corrosion protection from a thin passivating oxide film (approximately 10 nm thick), which forms spontaneously with surrounding oxygen. Titanium is considered the most biocompatible metal used for dental restorations produced with prostheses (44). Titanium and Ti alloys are advantageous in dental applications, notably because of biocompatibility, high strength, and corrosion resistance (45). Titanium is the most commonly used material for the fabrication of oral implants. This is supported by favourable mid- and long-term clinical outcomes (46).

According to the American Society for Testing and Materials (ASTM), there are six distinct types of titanium available as implant biomaterials. Amongst these six materials, there are four grades of commercially pure titanium (CpTi) and two titanium (Ti) alloys. The mechanical and physical properties of CpTi are different and are related chiefly to the oxygen residuals in the metal. The two alloys are Ti-6Al-4V and Ti-6Al-4V-ELI (extra low interstitial alloys). The commercially pure titanium materials are called pure Grade I, Grade II, Grade III and Grade IV titanium. Commercially pure titanium is also referred to as unalloyed titanium and usually contains some trace elements of carbon, oxygen, nitrogen and iron. Theses trace elements markedly improve the mechanical properties of pure titanium and are found in higher amounts from Grade I to Grade IV. Titanium alloys of interest to dentistry exist in three structural forms: alpha (α), beta (β) and alpha-beta. The alpha-beta combination alloy is the most commonly used for the fabrication of dental implants. This alloy consists of 6% aluminium and 4% vanadium (Ti-6Al-4V). Heat treatment of these alloys generating fine precipitation improves their strength, resulting in favourable mechanical and physical properties that make them excellent implant materials. They have a relatively low density, are strong and highly resistant to fatigue and corrosion. Although they are stiffer than bone, their modulus of elasticity is closer to bone than any other implant material, with the exception of pure titanium. This lower modulus of elasticity is desirable, as it results in a more favourable stress distribution at the bone-implant interface. Vanadium free α + β alloys, such as Ti-6Al-7Nb and Ti-5Al-2.5Fe, have been developed as implant materials because of toxicity concerns with vanadium (47).

2.3. Dental Ceramics

In dentistry, ceramics represents one of the four major classes of materials used for the reconstruction of decayed, damaged or missing teeth. Other three classes are metals, polymers, and composites. The word Ceramic is derived from the Greek word “keramos”, which literally means ‘burnt stuff’, but which has come to mean more specifically a material produced by burning or firing. In dentistry, ceramics are widely used for making artificial denture teeth, crowns, bridges, ceramic posts, abutments, and implants and veneers over metal substructure. Ceramics are characterized by their refractory nature, hardness, chemical inertness, biocompatibility and susceptibility to brittle fracture.

In 1789 a French dentist De Chemant patented the first porcelain tooth material. In 1808 Fonzi, an Italian dentist invented a "terrometallic" porcelain tooth that was held in place by a platinum pin or frame. Ash developed an improved version of the platinum tooth in 1837. Dr. Charles Land patented the first Ceramic crowns in 1903. Vita Zahnfabrik introduced the first commercial porcelain in 1963 (48).

2.3.1. Classification of dental ceramics

The ideal classification system of ceramic materials should be useful to giving clinical data about side to use the ceramic material (anterior or posterior) for which type of restoration (partial or full, short or long span) and how to cement it. Different classification systems have proposed according to clinical indication, composition, sensitivity to etching, methods of fabrication, firing temperatures, microstructure, fracture resistance and translucency (49).

Table 1: Classification of dental ceramics (48)

According to on the microstructure	Type 1: Glass-containing ceramics Type 2: Glass-containing ceramics with Reinforcing fillers Type 3: Crystalline-based ceramics Type 4: Polycrystalline ceramics
According to method of fabrication	1. Powder/liquid 2. Hot pressing 3. CAD/CAM
According to composition	1. Silicate ceramics 2. Oxide ceramics 3. Glass ceramics
According to the type	Feldspathic porcelain. Leucite – reinforced porcelaine, Aluminons porcelaine. Glass infiltrated alumina, Glass infiltrated zirconia. Glass ceramics.
According to firing temperature	Ultra-low fusing < 850°C Low fusing 850°C - 1100°C Medium Fusing 1101°C - 1300°C High fusing >1300°C
According to Substructure metal	Cast Metal, Swaged metal, Glass Ceramics. Sintered core ceramics and CAD-CAM porcelain. The various types of metals in metal ceramics include noble alloys, base metals. Pure metals and Base Metal alloys (nickel, chromium).
Based on reinforcing method	Reinforced ceramic core systems. Resin -bonded ceramics. Metal–ceramics

2.3.1.1. Microstructural classification of dental ceramics

The new approach to classifying ceramic restorative material in to three families. 1- Glass-matrix ceramics. 2- Polycrystalline ceramics. 3- resin-matrixceramics (49).

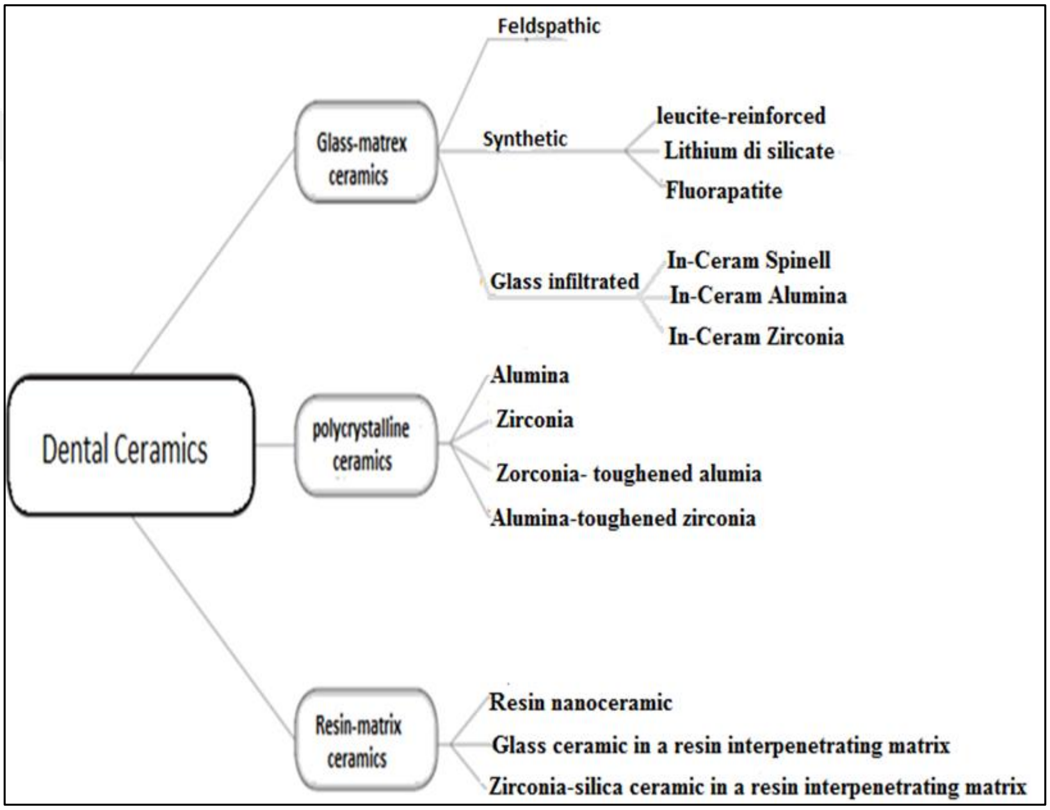

Figure 1: A new classification of ceramic amaterials (49)

2.3.1.1.1. Glass matrix ceramics

These are type of ceramics that contains glass matrix phase and at least one crystal phase is produced by controlled crystallization of glass (48). Glass-matrix ceramics are sensitive to acid etching by hydrofluoric acid (etchable ceramics) creating areas of micro retention, and it is subdivided into three sub group (49).

2.3.1.1.1.1. Feldspathic

This traditional group of ceramics materials composed of clay (hydrates aluminosilicate), quartz (silica), and feldspar (potassium and sodium alumina silicate). Potassium feldspar forms lucite crystals which increase the strength of restoration and lower the coefficient of thermal expansion for porclain veneering approximatly 10% below that of metal substructure. The feldspathic ceramics have low mechanical properties and low flexural strength 60–70 MPa. Therefore, it used as veneering materials for ceramic or metal substructure and as an esthetic materials bonded onto tooth structure (49).

2.3.1.1.1.2. Synthetic

It is a modification to feldspathic porcelain with addition of amounts of different crystals (leucite crystals or lithium disilicate crystals or fluor apatite crystals) into the glass matrix to enhance the mechanical properties for using as substructure matreial.

2.3.1.1.1.1.1. Leucite-reinforced glass–ceramics

Leucite has been widely used as a constituent of dental ceramics to modify the coefficient of thermal expansion which is important for ceramics to be fused onto metal. But in leucite reinforced ceramic system (IPS Empress), the leucite particles are incorporated to glass matrix to increase flexural strength. Newer generations of materials have much finer leucite crystals (10 μm to 20 μm) and even particle distribution throughout the glass. Therefore, these materials are less abrasive and have much higher flexural strengths up to120 Mpa. The most widely used version is the original pressable ceramic, IPS Empress (Ivoclar Vivadent). The leucite-reinforced glass–ceramics are most commonly used as veneer porcelains for metal-ceramic restorations (48).

2.3.1.1.1.2.2. Lithium disilicate glass-ceramics

Lithium disilicate is a dental ceramic that mimics the esthetics and strength of natural tooth structure (50). This type of dental glass-ceramic originally introduced by Ivoclar Vivadent as IPS Empress II (and later in the form of IPS e.max pressable and machinable ceramics). The lithium disilicate crystals represent about 70 % of the volume of glass-ceramic. It has microstructure consisting of numerous small plate-like crystals that are interlocking and randomly oriented which has a reinforcing effect on strength because, the needle-like crystals deflect cracks and arrest the propagation of cracks. The mechanical properties of this glass ceramic are far superior to that of the leucite glass ceramic, with a flexural strength in the region of 350– 450MPa and a fracture toughness approximately three times that of the leucite glass ceramic (48) .There is a possibility for its use in all-ceramic systems. It can be very trans lucent even with the high crystalline content. This is due to the relatively low refractive index of the lithium disilicate crystals. Lithium disilicate is etchable material due to the glassy phase which enhance the bonding properties (51). Initial clinical data for single restorations are excellant with this material, especially if it is bonded. Recently, lithium disilicate ceramic is now considered as one of the best restorative materials available for fabrication single unit indirect restorations (crown, onlays, inlay, veneer, hybrid implant abutments) whether for anterior or posterior area in the mouth. Also, the fabrication is done either by hot pressing technique or by CAD/CAM technique. Lithium disilicate is being increasingly used to replace zirconia (52) because of their biocopatibility, chemical durability, and the ability to replicate the optical characteristics of natural teeth. The already extensive indication range – from thin veneers (0.3 mm) and monolithic crowns to anterior and premolar bridges – has now been expanded to include hybrid abutment restorations. Recently, with IPS e.max Press fabrication of such restorations in combination with a titanium base (Ti base). Two different approaches are available for this purpose:1. hybrid abutments and 2. hybrid abutment crowns which combines the abutment and the monolithic crown in one piece. Both solutions show outstanding function, efficiency and esthetics. The durable bond to the Ti base is achieved by means of the self-curing Multilink® Hybrid Abutment luting composite (53). Before cementation of lithium disilicate restorations, one of the surface treatments either sandblast with 30- to 50-μm Al2O3 particles or etching with 9% hydrofluoric acid followed by suitable silane coating are recommended to ensure maximum bonding mechanism.

2.3.1.1.1.2.3. Fluorapatite Ceramics

A veneer porcelain made of fluorapatite glass. The shape and volume of fluorapatite crystals increase the flexural strength to approximately 360 MPa, or about three times that of Empress. This material can be translucent even with the high crystalline content which improves the optical properties of porcealin veneer.

2.3.1.1.1.3. Glass-infiltrated Ceramics

Aluminous porcelain contains a glass matrix phase and at least 35 vol% of alumina. It is a commonly used core ceramic and has a thin platinum foil when employed with all ceramic restorations. Aluminous core is stronger than feldspathic porcelain. The core substructure was sintered on a porous refractory die and then applying of slurry based molten glass on sintered coping or framework at 11000°C for about 4 hrs, the glass infiltrate into all the pores by capillary action to produce the dense more strength ceramic. The glass infiltrated ceramics can be fabricated by one of three core ceramics, In-Ceram Alumina, In-Ceram Zirconia and In-Ceram Spinell. They have adequate flexural strength and ability to be etched by acid etching (48).

2.3.1.1.1.3.1. InCeram Spinell (alumina-magnesia matrix)

It is the highest translucent with moderate strength, the flexural strengths are 350MPa, which can be used for anterior crowns (48).

2.3.1.1.1.3.2. InCeram Alumina (alumina matrix)

In-ceram alumina has very high strength and moderate translucent properties. In-ceram alumina flexural strength is 450 MPa and is used for anterior and posterior crowns.

2.3.1.1.1.3.3. InCeram Zirconia (alumina-zirconia matrix)

It İs a modification of In-ceram Alumina where parially stabilized zirconia oxide is dded to strengthen the ceramic. It very high strength and low translucent and is used for posterior bridges. In addition, these materials are supplied in a block form for producing milled restorations using a variety of machining system and the use of glass infiltrated ceramic is decrease due to increase the popularity of use of zirconia and lithium disilicate (49).

2.3.1.1.2. Polycrystalline ceramics

Solid-sintered monophase ceramics are formed by directly sintering crystals together without any intervening matrix to form a dense, air-free, glass-free, polycrystalline structure. Polycrystalline ceramics are not etchable and thus much more difficult to bond. Several processing techniques allow the fabrication of either solid-sintered aluminous oxide (alumina, Al2O3) or zirconium oxide (ZrO2) framework (48, 51).

2.3.1.1.2.1. Alumina

Fully dense polycrystalline material for dental applications was Procera® AllCeram alumina (Nobel Biocare, www.nobelbiocare.com), with a strength of approximately 600 MPa. The alumina powder is pressed and milled on a die and sintered at about 1600°C, leading to a dense coping but with approximately 20% shrinkage.

2.3.1.1.2.2. Zirconia

The use of zirconia has increased rapidly in the past few years. Partially stabilized zirconia with small amounts of other metal oxides allows production of reliable multiple-unit all-ceramic restorations for posterior teeth. Zirconia has unique physical characteristics that make it twice as strong and tough as alumina-based ceramics. Values for flexural strength range from approximately 900 MPa to 1100 Mpa. Fracture toughness, which has been reported between 8 MPa and 10 MPa for zirconia. This is significantly higher than other dental ceramics. Fracture toughness is a measure of a material's ability to resist crack growth. Zirconia has the apparent physical properties to be used for multiple-unit anterior and posterior FPDs. Clinical reports on zirconia have not shown any problem with the framework, but have shown the chipping and cracking of porcelain (51).

2.3.1.1.3. Resin-Matrix Ceramics

This type comprises materials with organic materix highly filled with ceramic particles. This materials closely simulate the modulus of elasticity of dentin, easier to milling and adjusting than glass-matrix ceramics or polycrystalline ceramics, also resin-matrix ceramic materials easy to repair or modification with composite resin. Resin-matrix ceramic composition varies substantially, but they are only fabricated for CAD/ CAM system. Recently, resin-matrix ceramic materials can be divided according to their inorganic composition into three subfamilies as follows: 1. Resin nano-ceramic (e.g., Lava Ultima, 3M).2. Glass ceramic in a resin matrix (e.g. Enamic, Vita). 3. Zirconia-silica ceramic in a resin matrix (49).

2.3.1.2. Classification of ceramic according to method of fabrication

Recent advances in ceramic processing methods have simplified the work of the dental technician and have allowed greater quality control for ceramic materials, which has increased their mechanical reliability. Ceramics having similar composition may be fabricated by different laboratory techniques, and each method of forming results in a different distribution of flaws, opportunity for depth of translucency, and accuracy of fit. These differences should be important to the clinician because they persist beyond the walls of the dental laboratory and affect clinical performance.

2.3.1.2.1. Powder/liquid system

This conventional construction of ceramic prostheses involves compaction, firing and glazing. The aim of condensation technique is to remove water to compact the powder particles then firing to drive off excess water to limit crack propagation then glazing is done to eliminate residual surface porosity (48). Ceramics fabricated by powder condensation have greater translucency than can be achieved using other methods, so these materials are usually applied as the esthetic veneer layers on stronger cores and frameworks (54).

2.3.1.2.2. Slip casting

A slip is a low viscosity mixture of ceramic powder particles suspended in a fluid (usually water). Slip casting involves forming a mold of the desired framework geometry and pouring a slip into the mold. The mold is made of a material (usually gypsum) that extracts some water from the slip into the wall and some of the powder particles in the slip become compacted against the walls of the mold forming a thin layer of green ceramic that is to become the framework. Then, the framework can be removed from the mold after partial sintering to improve the strength to a point where the framework can support its own weight. The resulting ceramic is very porous and must be either infiltrated with molten glass or fully sintered before veneering porcelain can be applied. Ceramics fabricated by slip casting can have higher fracture resistance than those produced by powder condensation because the strengthening crystalline particles form a continuous network throughout the framework. Use of this method in dentistry has been limited to one series of three products for glass infiltration (In-Ceram, Vita Zahnfabrik). The limited application of slip casting in dentistry is probably because the method requires a complicated series of steps, which provide a challenge to achieving accurate fit and may result in internal defects that weaken the material from incomplete glass infiltration.

2.3.1.2.3. Hot pressing

The lost wax method is used to fabricate molds for pressable dental ceramics. Pressable ceramics are available from manufacturers as prefabricated ingots made of crystalline particles distributed throughout a glassy material. The microstructure is similar that of powder porcelains, however, pressable ceramics do not contain much porosity and can have a higher crystalline content because the ingots are manufactured from non-porous glass ingots by applying a heat treatment that transforms some of the glass into crystals. This process can be expected to produce 1- a well-controlled and 2- homogeneous material. The pressable ingots are heated in the dental laboratory to a temperature at which they become a highly viscous liquid, and they are slowly pressed into the lost wax mold (40). Pressable ceramics maybe used for inlay, onlay, veneers, and single- unit crowns (34). Pressable veneering materials, such as IPS e.max ZirPress (Ivoclar-Vivadent) are available. The advantage of this technique is that it utilizes the experience that the lab technician already has in lost wax method with metal alloys .Pressable ceramic are categorized in to two generations including the first generation of heat-pressed dental ceramic contains leucite as reinforcing crystalline phase (IPS Empress 1) and the second generation is lithium disillicate- based(IPS Empress 2)[(55)]

2.3.1.2.3.1. IPS Empress1

It is a leucite glass ceramic with pressing temperature 1150-1180 ^{0}C. IPS Empress has a low flexural strength of 112±10 MPa limiting its use to single unit complete-coverage restorations in the anterior area [(55).]

2.3.1.2.3.2. IPS Empress 2

It is a lithium disilicate glass ceramic with pressing temperature 890-920 ^{0}C. Flexural strength of IPS Empress in the range of 400±40 MPa which is much higher than that of IPS Empress, increased flexural strength makes it suitable for the usage for fabrication of FPDs in the anterior region, and can extend to the second premolar [(55)].

More recently, IPS e.max Press (Lithium disilicate glass- ceramic ingot for the press technique was developed by Ivoclar–vivadent. It is available with different opacity HT, LT, MO, HO). In comparison with IPS Empress 2, it has better physical properties and improved esthetics. Accordıng to manufacture the IPS e.max Press material used to fabricate inlays, onlays, veneers, partial crowns, anterior and posterior crowns, short span anterior bridges that can be extend to premolars, telescope primary crowns, implant abutments and implant restorations [(55)].

2.3.1.2.4. Computer-aided design/computer-aided manufacturing (CAD-CAM)

As presseble ceramics, CAD-CAM are available as prefabricated glass-ceramic ingots. These ingots are milled or cut by computer-controlled tools. An optical impression is taken for the preparation by a special scanner the image is then transferred to the system’s software, then the software designs the restoration and sends the data to the computer controlled milling machine that grinds the ceramic block according to the desired shape. Many of materials available for the CAD/CAM technology such as, silica based ceramics: IPS e.max-CAD. Infiltration ceramics: Vita In-Ceram, Oxide high performance ceramics and blocks of aluminum oxide and zirconium oxide (56).

2.3.1.3. Pretreatment for ceramics based on their classification

Dental ceramics, because of their differences in composition and phases, therefore require different pretreatment procedures. Silica-based ceramics will require either etching with hydrofluoric acid or sandblasting and subsequent silanization to improve adhesion to the resin cement. Hydrofluoric acids (HF) roughen the internal surface of the restoration. They are available in varying concentrations from 2.5 to 10 %, and etching time is usually 2–3 minutes. Etching ceramic with hydrofluoric acid renders the surface microscopically porous, increases the surface energy resulting in a micro-retentive surface. Care should be taken not to over-etch the porcelain with hydrofluoric acid as it can weaken the bond between the ceramic and resin cement. After HF etching, a white residue sometimes forms on the surface of the porcelain. This white residue is a potential contaminant and should be removed prior to silane application. Recommended methods of removing this residue include immersing in an ultrasonic cleaner for 5 min, steam cleaning, or using an alcohol solution. Silane-coupling agents, or simply silane, ensure a good bond between the hydroxyl groups of the ceramic and the organic portion of the resin cement. The silane is applied on the internal ceramic surface and then air-dried. There is no consensus on the duration of silane application as it may range from 5 min to 2 h. The usual application time is between 60 and 90 s. Non-silica-based ceramics such as alumina and zirconia have polycrystalline phase and should not be etched as they are highly resistant to chemical attack from HF or silanated as it might destroy the crystalline structure and weaken the material. The preferred pretreatments for alumina or aluminum oxide ceramics include (1) airborne abrasion with 50–110 μm aluminum oxide particles at 2.5 bars, (2) use of an MDP-containing resin cement (Panavia 21, Kuraray, Japan; Single Bond Universal (3 M Espe, Germany), or (3) silicoating through tribochemical surface treatment (Rocatec, 3 M Espe, Germany) followed by application of a conventional bis-GMA resin cement (57).

Table 2: Surface treatments for the different types of porcelain (57)

Type of ceramic	**Pre-Surface treatment**
Feldspathic porcelain Leucite-reinforced ceramic Lithium disilicate	Hydrofluoric acid 2.5–10 % for 2–3 min or sandblasting/air abrasion or sandblasting + HF acid etching with application of silane following manufacturer’s instructions
Alumina/aluminum oxide	1. Airborne particle abrasion (APA) using 50–110 μ AlO 2 at 2.5 bars or 2.Use an MDP containing resin cement and primer (Panavia F 2.0, Universal Bond) or
Zirconia/zirconium oxide	3. Silicacoating (tribochemical surface treatment) 4. APA or silica coating + use an MDP containing resin cement CEREC in Lab 5. Use a phosphoric acid monomer containing primer (Z-Primer, Metal/Zirconia Primer, AZ Primer) without silane application

2.3.1.4. Cementation of all ceramic restorations

The protocol used for cementation of all ceramic restoration can be essential for success (58). Clinicians can effectively etch silica-based all-ceramic for adhesive bonding. The clinical life span of such all-ceramic restoration significantly increased when this protocol used. Zirconia and alumina-based all ceramic materials can’t be etched and bonded (56).

2.4. Dental cements

Luting agents are used like glue to retain the metallic, ceramic and composite crowns, bridges inlays and onlays permanently. Ideally, the luting cement should be biocompatible, have sufficient light transparency and strong enough against mastication forces. Luting mechanisms of cements are three types; chemical, mechanical and micromechanical. Retention of restoration is obtained, depending on the quality of applied cement, through combining two or three of these mechanisms (59). Each cement type has different physical, mechanical and biological features arising from its own chemical structure. That is why one single cement type alone is not sufficient for daily clinical applications. To achieve a clinical success, any clinician is expected to be aware of the qualities, advantages and disadvantages of each type of cement and conduct their clinical applications (60). Modern dentistry has a wide variety of application products differing from each other in content and physical attributions (59). Therefore, it may pose difficulty for dentists to make a choice amongst so many alternative products (61). O’Brien classified dental cements by matrix bond type (ie, phosphate, phenolate, polycarboxylate, resin and resin modified glass ionomer). Donovan simply divided cements into conventional (zinc phosphate, polycarboxylate, glass-ionomer) and contemporary (resin-modified glass-ionomers, resin) based on knowledge and experience using these materials. Resin cements as Variolink II (Ivoclar, Vivadent) chemically bond to etched, silane-treated porcelain. Resin cements can be activated chemically or via visible light or by both chemical and light (dual). Based on a good number of laboratory and clinical researches, it can be suggested that resin cements are the best choice for the cementation of full ceramic restorations. Furthermore, resin cements can form a better bonding with metal alloys sanded via micromechanical retention.

Today many of the resins that are termed as adhesive are not actually with adhesive attributions. Only adhesive resins with monomers containing 4META and MDP have adhesive quality. The first product marketed, Panavia, contained the bifunctional adhesive monomer MDP (10-methacryloyloxydecyl dihydrogen phosphate) and was a powder-liquid system. Bond strength to etched base metal greatly exceeded that to tooth and Panavia quickly became the luting agent of choice for resin retained fixed partial dentures. Conventional cementation and adhesive cementation are, let alone being conflicting, complementing each other. The choice should be based on the type and design of planned restoration because none of the present products possesses all qualities of an ideal luting agent. Each cement type has different physical, mechanical and biological features arising from its own chemical structure. That is why one single cement type alone is not sufficient for daily clinical applications. To achieve a clinical success, any clinician is expected to be aware of the qualities, advantages and disadvantages of each type of cement and conduct their clinical applications (62).

2.5. Bond strength measurement tests

An understanding of the bonding mechanism is essential for successful metal-ceramic restorations. Various tests have been designed and selected by re-searchers to evaluate metal-ceramic bond strengths. These tests can be classified according to the nature of stresses created such as shear, tension, combination of shear and tension, flexure, and torsion test designs (63).

2.5.1. Shear bond strength test

In a shear bond test, two materials are connected via bonding agent and loaded in shear until failure of bond occurs. It is the most common method to evaluate the quality of the adhesive bond simply because it is considered as simple to perform, clear test protocol, and rapid production of test results (64). The values of bond strength are calculated by dividing the maximum applied force leads to failure of the bond by the bonded cross-sectional area of the bonding interface (65). Although the shear bond strength test is simpler than the tensile bond strength test, the former has a limitation of measuring the cohesive strength of the adhered (or sometimes substrate) rather than the true bond strength of the bonding interface. Therefore, the shear bond strength test does not simulate the clinical mode of failure, which is mainly adhesive failure. When the main failure mode is cohesive in a bond strength test, the bond strength values have no definite clinical meaning. Furthermore, shear bond strength tests require large specimen sizes, especially in the case of ceramic materials may result in increased structural flaws that may lead to premature failure of the test specimen before the maximum bond strength levels are reached (66).

2.5.2. Tensile bond test

In this test, load will be exerted on either side of the test specimens. The specimen can be held by active or passive gripping methods. In the tensile test, stresses are far more homogenous across the interface than shear. Therefore, the maximum stress values are much closer to the nominal strength. It can measure the bond strength of cements to hard materials such as ceramics and metal alloys (64).

2.5.3. Bend tests (flexure)

The bend test with three- or four- point loading was selected by Lavine and Custer, Caputo et al., J and O’Brien and Craig.A flat strip of metal was used with porcelain fired on the tensile face, which was then tested for transverse strength (modulus of rupture). Transverse strength is breaking strength in a non-ductile solid, such as porcelain, measured by bending. This is usually identified as bend strength on flexural strength. Finite element stress (hypothetical) analysis demonstrated higher tensile stresses compared with shear stresses, creating a greater probability of tensile failures. Tensile stresses could be either perpendicular or parallel to a metal-ceramic interface. The relative importance of each tensile stress directional component remains unknown. Four-point loading tests were successfully used to separate porcelain from metal when interfacial failures consistently developed at load points with microscopically clean separation between porcelain and metal. Four-point loadings were also easy to fabricate, required no special equipment for testing, and thicknesses of porcelain and metal simulated clinical conditions (63). Bend tests were subject to criticism because maximal tensile stresses were created at the surface of porcelain and resulted in predictable tensile failures. The major difficulty with bend tests was related to analysis of stress states that were present. The validity of these tests to evaluate different alloys has been questioned because ceramic breakage depended on the modulus of elasticity of the metal tested. An alloy with an elevated modulus of elasticity would resist bending to a greater extent, creating a higher bond. Therefore, it becomes suspect as to whether the bond or the modulus of elasticity of the metal is the characteristic actually tested (63).

3. MATERIAL AND METHODS

In this study 40 titanium alloy (Ti6Al4V; Eisenbacher Dentalwaren; Germany) disk-shaped (6.6 mm diameter and 4 mm thickness) were fabricated using CAD/ CAM technology and identical number and size of lithium disilicate disks (IPS e.max P ress;Ivoclar Vivadent AG) were fabricated by heat pressing technique.

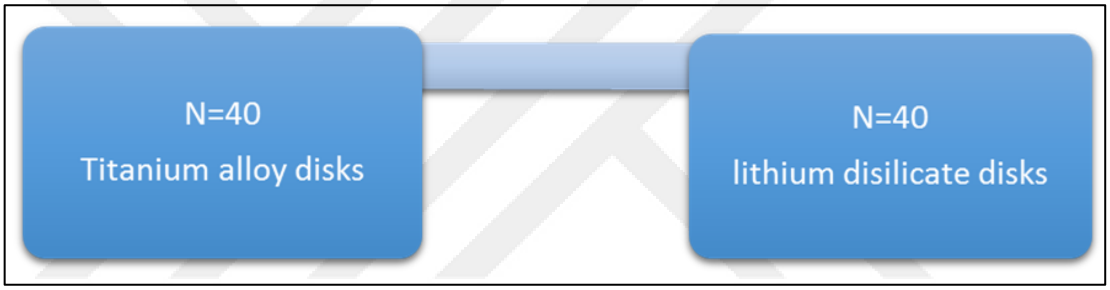

Figure 2: Flow- chart illustrate the total number and materials of samples

3.1. Fabrication of titanium disks

We used the CAD-CAM system to fabricate the titanium disks. Therefore, the size of the disks were designed on computer with 6.6 mm diameter and 4.0 mm thickness .Then titanium disks were cut from CAD/CAM titanium blocks (Kera Ti 5-Disk,) in; Eisenbacher Dentalwaren; Germany) with CAD/CAM machine.

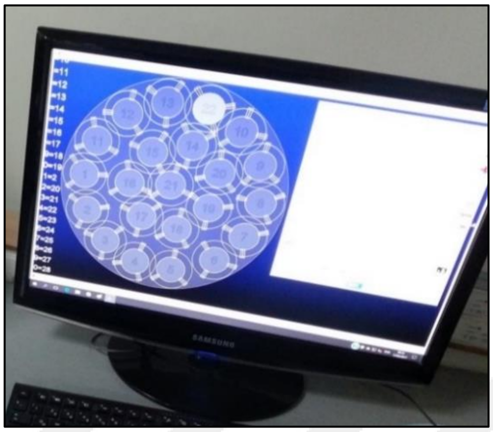

Figure 3: Design of titanium disks with cad /cam system

Figure 4: Milling of titanium disks in cad/cam machine

Figure 5: Titanium disks after cutting

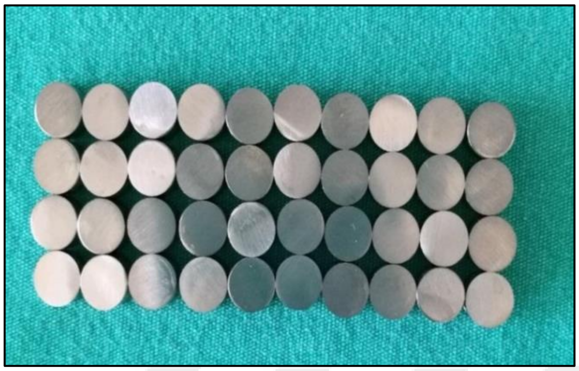

Figure 6: Forty titanium alloy samples

3.2. Fabrication of lithium disilicate glass ceramics disks

Forty disks (6.6 mm diameter × 4.0 mm thickness) of IPS e.max press ceramic (Ivoclar; Vivadent) shade LTA1 were fabricated in accordance of manufacture`s instruction. After the disk’s sizes were designed on computer in cad/ cam system, laboratory procedures were began by cutting the wax blocks (Kronenwachs;Bego ;Germany) to make round wax patterns. Then the wax disks were subjected to spruing then attached to a muffle base (five per muffle) with a surrounding paper cylinder. Wax patterns were invested with phosphate-based material (IPS Press Vest Speed, Ivoclar; Vivadent). Wax was eliminated in an automatic furnace at 850 °C for 1 h. IPS e.max Press ceramic ingots were pressed into the molds in an automatic press furnace (EP 600; Ivoclar Vivadent). After cooling, specimens were divested and submitted to wet polishing.

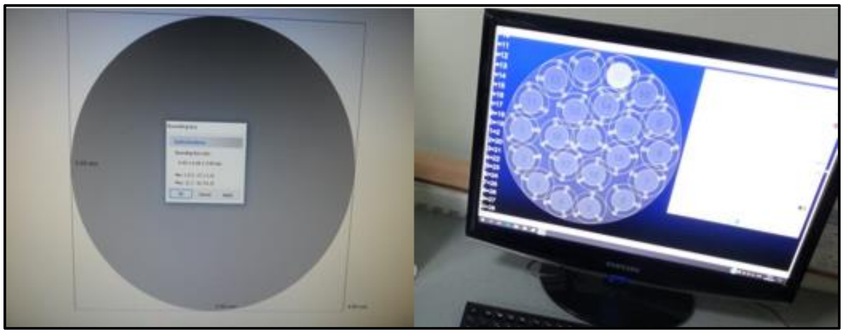

Figure 7: Design of the lithium disilicate disks in cad/cam system

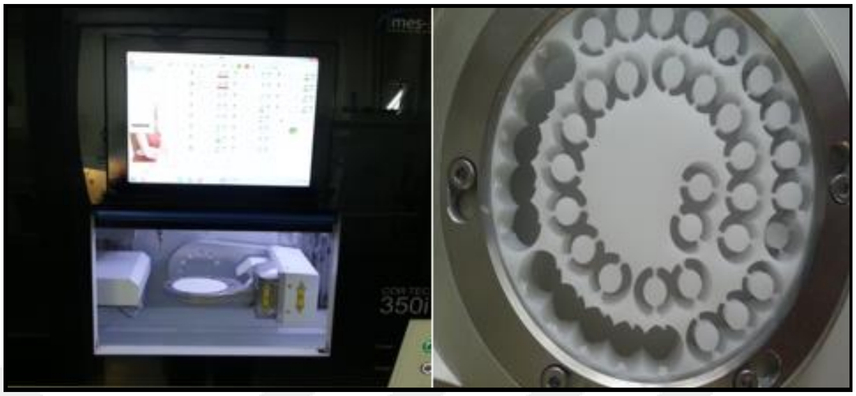

Figure 8: Cutting of wax by cad/cam system

Figure 9: Spruing of wax disks

Figure 10: Automatic furnace for wax elimination

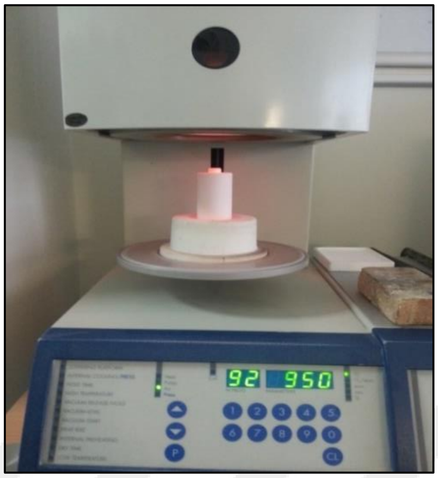

Figure 11: Automatic press furnace for pressing the IPS e. max press ingots

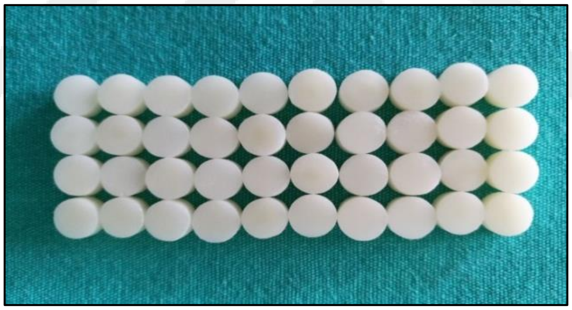

Figure 12: Forty lithium disilicate samples

After specimen fabrication, all bonding surfaces of both titanium and lithium disillicate disks were polished with silicon carbide paper by a polishing machine (Buehler Phoenix BETA Grinder Polisher) with wet silicon carbide paper, grinding with 600- and 1000-grit under water cooling.

Figure 13: Polishing machine

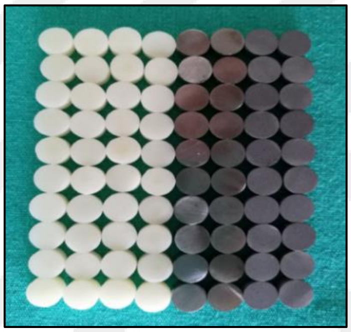

Figure 14: Both titanium & ceramic disks after polishing

Table 3: Materials tested in this study

Brand	Manufacturer	Lot number
Ti6Al4V	Eisenbacher Dentalwaren; Germany	Ti13-12
IPS.max press	Ivoclar vivadent AG; Liechtenstein	FL-9494
MultilinkHybrid Abutment	Ivoclar vivadent AG; Liechtenstein	V17072
Panavia SA cement Plus Automix	Kuraray; Germany	880109
Porcelain etchant (9.5%HF)	Bisco; USA	1700000591
IPS Ceramic Etching Gel	Ivoclar vivadent AG; Liechtenstein	V31521

3.3. Surface treatments& cement type

According to surface treatment applied with combination of two types of cements for cementation procedures titanium disks were divided into 4 groups (n=4)

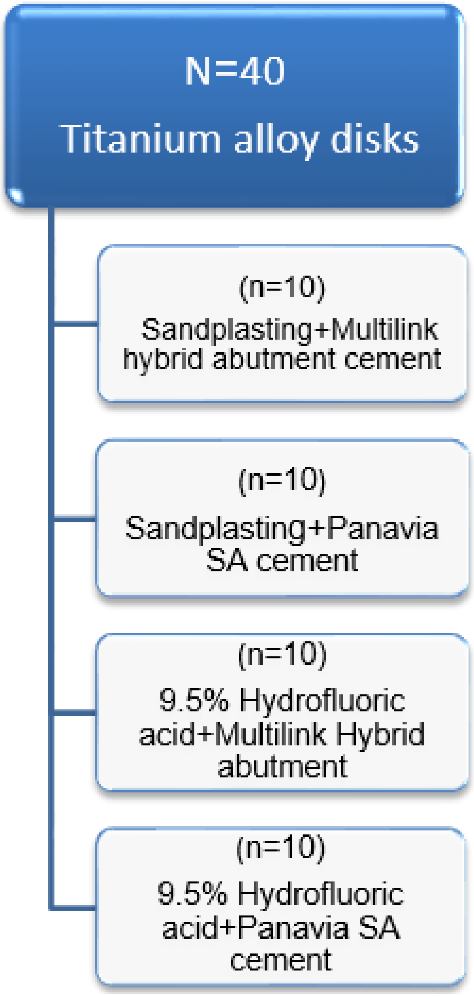

Figure 15: flow-chart illustrate titanium groups in the test

Group 1: Sandblasting with 50-µm alumina at 0.4-MPa pressure for 10 seconds at 20-mm distance according to manufacturer's instruction + Multilink hybrid abutments cements (Ivoclar; Vivadent)

Group 2: Sandblasting with 50-µm alumina at 0.4-MPa pressure for 10 seconds 20-mm distance according to manufacturer's instruction + Panavia SA cement (Kuraray).

Group 3: Etching with 9.5% Hydrofluoric acid (Bisco) for 90 seconds + Multilink hybrid abutments cement (Ivoclar; Vivadent).

Group 4: Etching with 9.5% Hydrofluoric acid (Bisco) for 90 seconds + Panavia SA cement (Kuraray).

For all groups, the lithium disilicate glass-ceramic disks received chemical etching with 4.5%Hydrofluoric acid (IPS Ceramic Etching Gel; Ivoclar Vivadent AG) for 20 seconds and were rinsed and air- dried before the cementation procedures.

3.3.1. Sandblasting procedure

The bonding surfaces of group 1 and group 2 totally 20 titanium disks were sandblasted with 50-µm alumina at 0.4-MPa pressure for 10 seconds at 20-mm distance according to manufacturer's instruction in (Renfert) Sandplaster device and were ultrasonically cleaned for 5 minutes in ultrasonic cleaning unit (Alex) followed by air- drying before the cementation procedures.

Figure 16: Sandblasting device

Figure 17: Ultrasonic cleaning unit

Figure 18: The Sandblasted Ti alloy disks

3.3.2. Etching with 9.5% Hydrofluoric acid

The bonding surfaces of group 3 and group 4 totally 20 titanium disks were etched with 9.5% Hydrofluoric acid gel (Bisco) for 90 seconds which are not currently recommended by the manufacturers of the components then rinsed and dried before the cementation process according to manufacturer’s instructions.

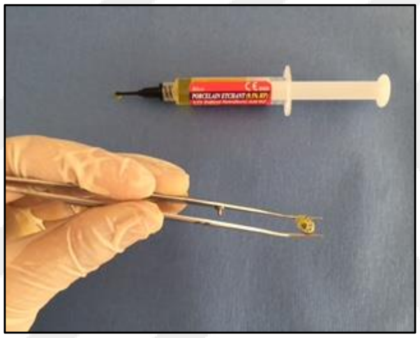

Figure 19: 9.5% HF acid gel application

3.3.3. Etching with 4.5 %Hydrofluoric acid of lithium disilicate disks

For all the groups the bonding surfaces of the lithium disillicate glass-ceramic disks totally 40 disks were chemical etched with 4.5% HF(IPS Ceramic Etching Gel; Ivoclar Vivadent AG) for 20 seconds and were rinsed for 90 seconds and air- dried according to manufacturer’s instructions before the cementation procedures.

Figure 20: 4.5%Hf acid application

3.4. The cementation procedures

Two types of cements were applied according to the manufacturer's instructions (1. Multilink hybrid abutment cement .2. Panavia SA cement plus automix). To standardize the cementation procedures an especially epoxy glass device with hole (6.7×7.0 mm) was designed to put the cemented disks in the correct position with application of 5 KG (50 N) load during the cement polumerization, to simulate the bite force in the mouth.

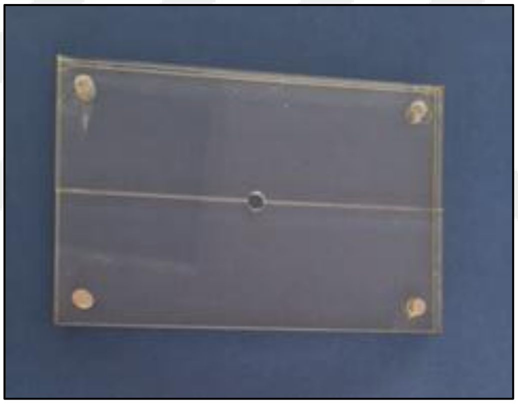

Figure 21: Epoxy glass device

3.4.1. Multilink hybrid abutment cement

For group 1 & group 3, the bonding surfaces of titanium disks and lithium disilicate glass ceramic were cemented with auto-polymerizing resin cement (Multilink Hybrid Abutment;Ivoclar Vivadent AG) .A thin layer of Multilink hybrid abutment cement was applied directly from the mixing syringe to the titanium and the lithium disilicate bonding surfaces , then the specimens were positioned together in the hole of the device with application of a light finger pressure for 10 seconds then the excess cement was gently removed with a micro brush and a load of 5 kilogram was applied during auto polymerization for 10 minutes at room temperature.

Figure 22: Multilink hybrid abutment cement application

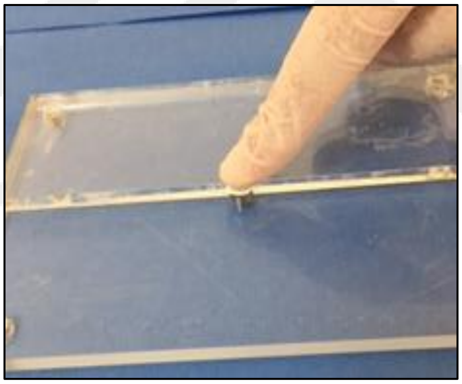

Figure 23: Finger pressure application

Figure 24: 5 kg load application

3.4.2. Panavia SA cement Plus Automix

For group 2 & group 4, the bonding surfaces of titanium diks and lithium disilicate glass ceramic were cemented with dual-polymerizing resin cement (Panavia SA cement Plus Automix ;Kuraray); by applying thin layer directly from the mixing syringe to the clean bonding surfaces .Then the specimens were lightly pressed together and held in the hole of the device then light cured for 5 seconds by BLUE LED light curing unit with application of a light finger pressure for 10 seconds and the excess cement was gently removed with a micro brush. After that, a load of 5 kilogram was applied during photo-polymerization for 10 seconds per side. Cemented samples were removed from the device then the light cured once again about 10 seconds.

Figure 25: Panavia SA cement application

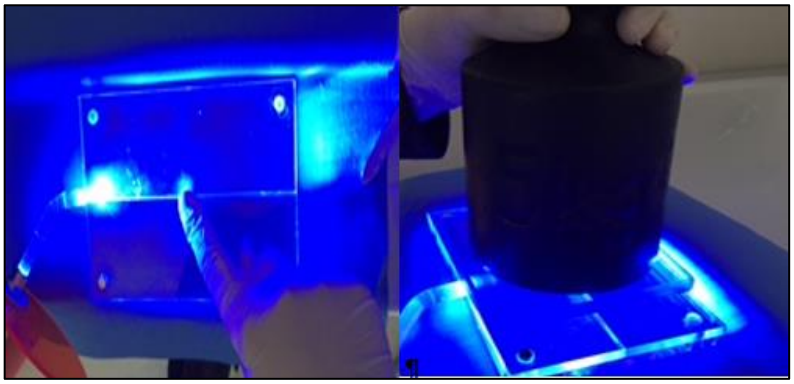

Figure 26: Light curing with load application

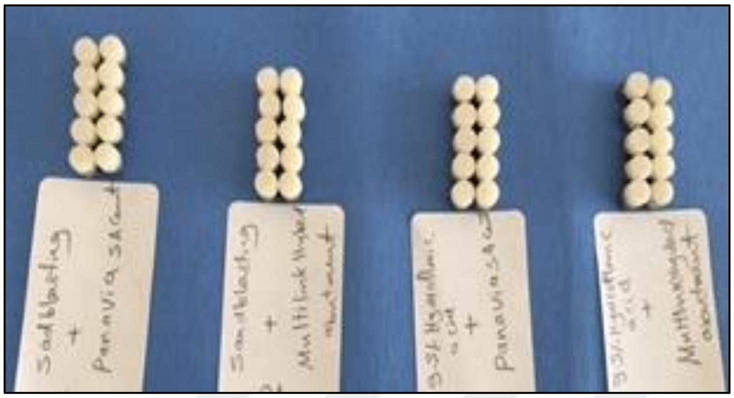

Figure 27: All the cemented samples groups

3.5. Thermocycling

The cemented samples were stored in distilled water at 37°C for 24 hours before the thermal cycling in thermocycling machine with water temperature between 5°C and 55°C for 5000cycles and a 15-second dwell time and a transfer time from one bath to the others of 10 seconds.

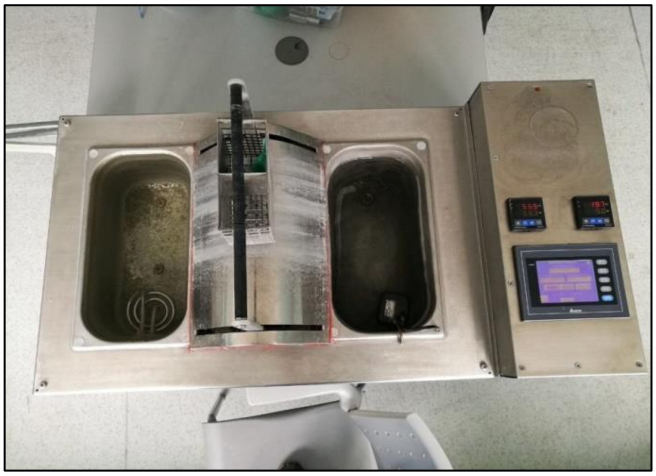

Figure 28: Thermocycling machine

3.6. Shear bond stress test

All the bonded samples were embedded in blocks of chemical cured acrylic resin (Imicryl, TURKEY) by using a specially designed steel mold (12mm depth×20mm diameter) to facilitate clamping on the universal testing machine.

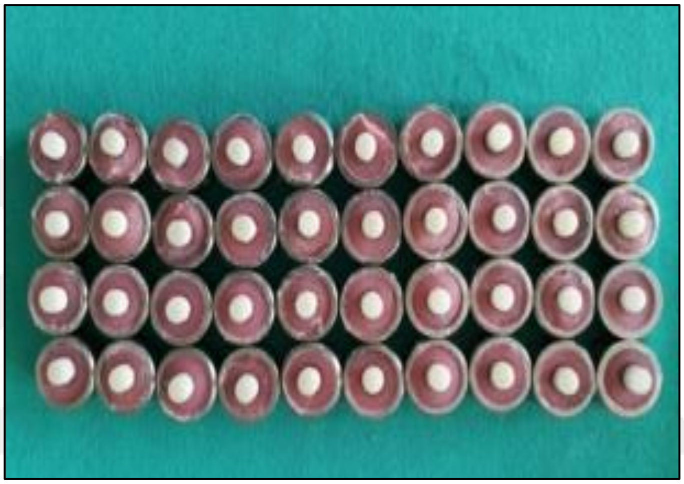

Figure 29: Fixing the cemented disks in steel mold

Shear bond testing of all groups was carried out by using of a universal testing machine (Instron 3345 model, USA) at a crosshead speed of 1mm/min. A knife-edge blade apparatus was near as soon as possible and parallel to the titanium and the lithium disilicte samples interfaces. The shear de bonding forces were registered in Newton (N). The failure loads (N) were divided by the bonding areas (mm^2), and then the shear de-bonding forces were changed into MPa.

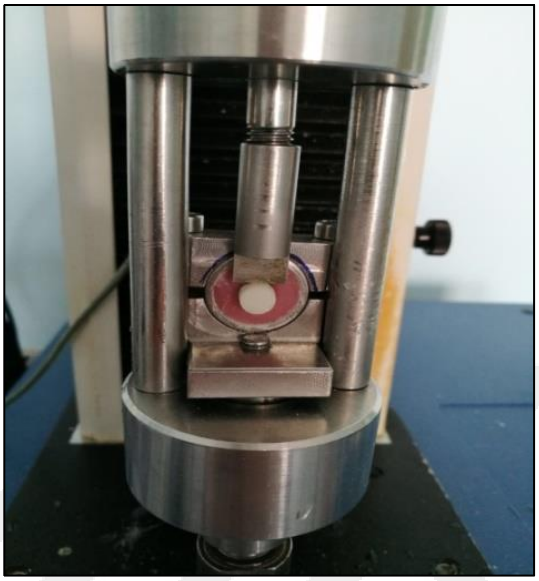

Figure 30: Universal testing machine

3.7. Statistical analysis

Statistical calculations were performed with (Number Cruncher Statistical System) 2007 Statistical Software (Utah, USA) program for Windows. Besides standard descriptive statistical calculations (mean and standard deviation), The four groups were compared using one way ANOVA followed by Tukey's post Hoc test, unpaired t test was used in the comparison of two groups. Statistical significance level was established at $p<0.05$.

4. RESULTS

The shear bond strength is reported in Mega pascal (Mpa). The Mean, SD, Minimum and Maximum values for all the groups in (MPa) by One-Way ANOVA test are shown in Table 4. The data were analyzed for significant differences between all the groups using a one-way analysis of variance (ANOVA) with $p < 0.05$, and are shown in Table 5. Differences between two groups were determined by Unpaired test and are shown in Table 6. Figure: 31 represents stock-chart for mean shear bond strength of all groups in Mpa.

Table 4: The Mean, SD, Minimum and Maximum values for all the groups in (MPa)

One-Way ANOVA	**All the tested groups**	**N**	**Mean±SD**	**Minimum**	**Maximum**
Shear bond Strength (MPa)	**Group1** Sandblasting/Multilink Hybrid Abutment	10	15.91±3.23	11.43	19.96
	Group2 Sandblasting/Panavia SA cement Plus Automix	10	13.48±1.65	11.14	16.29
	Group 3 9.5% Hydrofluoric Acid/Multilink Hybrid Abutment cement	10	5.35±1.12	3.89	7.06
	Group 4 9.5% Hydrofluoric Acid/Panavia SA cement plus Automix	10	3.89±0.59	3.16	5.38

The mean SBS values [Mpa] for Group 1 (Sandblastings/Multilink hybrid abutment cement) is 15.91±3.23 , Group 2 (Sandblasting / Panavia SA cement Plus Automix) is 13.48±1,65 , Group 3 (9.5%Hydrofluoric Acid/ Multilink Hybrid Abutment cement) is 5.35±1.12 and Group 4 (9.5%Hydrofluoric Acid/ Panavia SA cement plus Automix) is 3.89±0,59.

Table 5: Comparison shear bond strengths between all the groups (Mpa)

Tukey Multiple Comparison Test	**P**
Group 1/ Group 2 Sandblasting Multilink Hybrid Abutment Cement/Sandblasting Panavia SA cement plus Automix.	0.036
Group 1/ Group 3 Sandblasting Multilink Hybrid Abutment Cement/9.5% Hydrofluoric Acid Multilink Hybrid Abutment cement	**0.0001**
Group 1/ Group 4 Sandblasting Multilink Hybrid Abutment Cement/9.5% Hydrofluoric acid Panavia SA cement plus Automix	**0.0001**
Group 2/ Group 3 Sandblasting Panavia SA cement plus Automix/9.5% Hydrofluoric Acid Multilink Hybrid Abutment cement	**0.0001**
Group 2/ Group 4 Sandblasting Panavia SA cement plus Automix/9.5% Hydrofluoric acid Panavia SA cement plus Automix	**0.0001**
Group 3/ Group 4 9.5% Hydrofluoric Acid Multilink Hybrid Abutment cement/9.5% Hydrofluoric acid Panavia SA cement plus Automix	0.046

There is no a statistically significant difference between the mean Shear Bond strength of Group 1 sandblasting/Multilink hybrid abutment cement and Group 2 sandblasting/Panavia SA cement plus Automix (P=0.036). There is statistically significant difference between Group1 Sandblasting/Multilink Hybrid Abutment Cement and Group 3 hydrofluoric Acid/Multilink hybrid abutment cement (P =0.0001). There is statistically significant difference between Group1 sandblasting/Multilink hybrid abutment cement and Group 4 hydrofluoric acid/Panavia SA cement plus Automix (P =0.0001). There is statistically significant difference between shear bond strength of Group 2 sandblasting/Panavia SA cement plus automix and Group 3 Hydrofluoric Acid /Multilink hybrid abutment cement (P =0.0001). There is statistically significant difference between shear bond strength of Group 2 sandblasting/Panavia SA cement plus automix and Group 4 hydrofluoric acid/Panavia SA cement plus Automix (P =0.0001). There is no a statistically significant difference between Group 3 Hydrofluoric Acid /Multilink hybrid abutment cement and Group 4 Hydrofluoric acid Panavia SA cement plus Automix (P=0.046).

Table 6: Unpaired test showed the differences between the two groups

Unpaired t test	**Multilink Hybrid Abutment cement**	**Panavia SA Cement plus automix**	**P**
Sandblasting	15.91±3.23	13.48±1,65	0.002
9.5% Hydrofluoric acid	5.35±1,12	3.9±0.6	0.048
P	**0.0001**	**0.0001**	

Regarding to air-brone particle abrasion and as surface treatment for titanium samples there is no a significant difference between the two cement types (P=0,002). Also for Hydrofluoric acid as surface conditioning for titanium specimens there is no a significant difference between whether Multilink hybrid abutment cement or Panavia SA Cement plus automix (P=0.048). On the other hand, there is a significant difference between the two surface treatments types of titanium samples within the same cement type (p=0.0001). In other words, sandblasting multilink hybrid abutment is higher than hydrofluoric acid multilink hybrid abutment (p=0.001) Also for sandblasting and panavia cement is higher than hydrofluoric acid etching and Panavia application (p=0.0001).

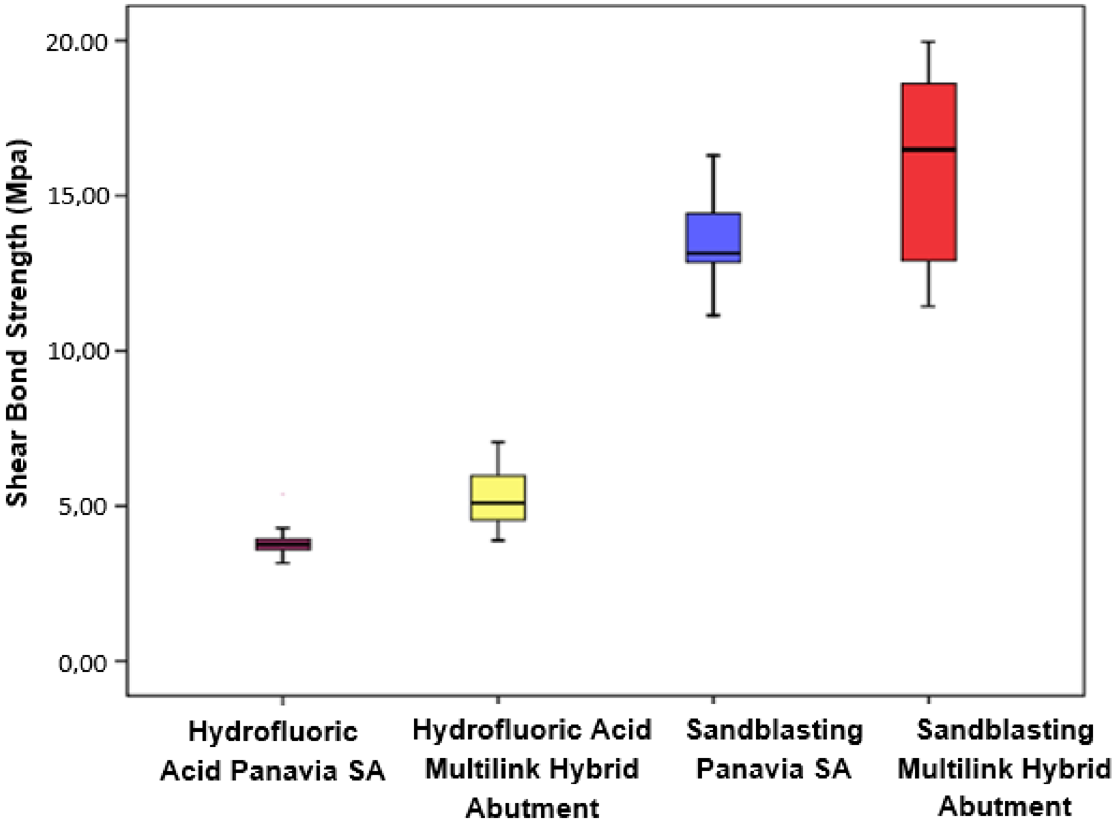

Figure 31: Stock-chart reprents mean shear bond strength of all groups in Mpa

5. DISCUSSION

Dental implants are widely used by specialists and general practitioners, and many new products are on the market. As such, demand for products that have longevity and are aesthetically acceptable is an important objective for the practitioner in selecting products to use. Implant abutment is the prosthetic portion of a dental implant restoration that connects to the implant fixture and serves to support and/or retain a prosthesis which made of metal or ceramic materials. Since ceramic implant abutments made their appearance in the 1990s, many studies have attempted to compare the reliability of ceramics to metal and evaluate the long term success and possible complications, considering patients' demands for aesthetics along with the increasing amount of new materials available (67).

Recently, the role of an abutment has expanded to include support of the soft tissue emergence and submergence profiles of a dental implant crown, as well to provide base shades at the cervical aspect of single tooth and multiple implant prostheses to allow for better esthetic prosthetic success. Such demands have led to the development of modern custom abutment fabrication techniques using titanium and ceramic. There are many options exist for implant abutments in the esthetic zone, one of the most abutment design is two- piece abutment or hybrid abutment which consists of metal base as Ti base or Ti abutment and ceramic segment bonded to each other by cementation procedures. Hybrid abutment can achieve a high strength due to the metal internal connection with the implant and produces a natural appearance near the root and transition area to the crown, which is especially important for anterior implant treatments. The long-term success of a dental implant is determined by several important factors one of them the mechanical properties of the prosthesis (68). Therefore, implant abutment materials should have appropriate strength as well as good esthetics. The use of lithium disilicate glass- ceramics in combination with titanium alloy to form hybrid type abutments and restorations for implants is likely to increase, primarily to improve the esthetics of the implant restoration. Besides esthetic properties of lithium disilicate glass ceramics, a high strength value of lithium disilicate ceramic bonding to a titanium- based insert of implants is essential. The connection of these two materials is a determinant of long term success and this depended mainly on the type of cement and the pre-surface treatments. The resin cements are the newest type of cements for indirect restorations, and they have the ability to bond to the tooth structure and the internal surface of the restoration. Resin cements are composed of the same basic component as the composite restorative material but with lower concentration of filler particles (Simon and Darnell 2012). These cements have higher compressive, flexural, and tensile strength than the conventional cements and can be used for almost any type of restoration and restoration material. One study reported that Using chemo-mechanical bonding systems, ie, silica-coating systems or modified composites with adhesive monomers, resulted in 2 to 2.5 times increased bond strength to titanium compared with the bond strength of a conventional bisphenol-Aglycidyl methacrylate composite (69). These cements however are more complex than the conventional cements and are highly technique sensitive. To maximize the properties of resin cements, a clear understanding of the factors that affect its clinical performance is of paramount importance. These factors are interrelated. The most important factor affecting the success of resin cements is the bond strength of the resin cement. Bond strength in turn is affected by pretreatment procedures, the depth of cure and degree of polymerization of the resin cement (70). The bonding between titanium alloy and the resin materials has been improved by using different methods which are called surface modifications including sandblasting, silica coating, chemical etching, electrolytic etching, plasma exposure and primer application (71). These procedures produce micromechanical interlocking or chemical bonding or both of them. Airborne-particle abrasion (APA) can be used as a surface treatment for metal alloys and ceramics (72). There are various parameters in airborne-particle abrasion with alumina the first parameter is the grain size which starts from 25 to 250 µm, the second parameter is the propulsion pressure starts from 0.05 to 0.45 MPa and the third is the distance from the nozzle to the specimen is ranged from 5 to 20 mm and the last one is the time of exposure is ranged from 5 to 30 seconds (73, 74)

Giachetti et al (75) analyzed the morphology of sandblasted titanium surface with different sizes of alumina particles by using the scanning electron microscopy. The result showed that the surface treated with 50µm alumina particles presented rough and irregular surface where the resin can penetrate. It has been reported that the sandblasting procedure makes the alloy-water contact angle more smaller and greater wettability and creates a roughened surface, and provide a great surface area for the bond (76). Kern and Thompson evaluated the surface morphology of sandblasted titanium alloy. They found that while most of the alumina was firmly embedded into the titanium surface any loose particles should be removed by using ultrasonic cleaning before the application of resin that contains chemically active monomers because these loose alumina particles can make the interfacial resin bond more weak (77).

Hydrofluoric acid is a promising alternative to sandblasting for the surface treatment of titanium substrate in the titanium-porcelain bonding system. Guilherme et al (78) evaluated the effect of sandblasting with 50 µm and hydrofluoric acid with different concentrations (5 %, 9.5%), and different etching times (30s, 90s). It was concluded that when 9.5% HF etching for 30 seconds was applied and gave the highest bond strength similar results to airborne-particle abrasion. As hydrofluoric acid etching is more controllable, requires less equipments, and is easier method than sandblasting, this technique may be a useful alternative for the clinician and the dental laboratory technician .

For lithium disilicate glass ceramics surface treatment, etching with hydrofluoric acid is recommended, the acid reacts with the glass matrix that contains silica and forms hexa-fluorosilicates. This glass matrix is selectively removed and the crystalline structure is exposed. As a result, the surface of the ceramic becomes rough, which is expected for micromechanical retention on the ceramic surface (79). This roughly etched surface also helps to provide more surface energy prior to combining with the silane solution (80). In vitro studies have reported positive effects of hydrofluoric acid (HF) etching on the strength of glasses by removing or stabilizing surface defects and on surface topography increasing roughness for adhesive bonding. Della Bona et al (81) suggested that etching mechanisms change according to the type of etchant and etching time, and the ceramic microstructure and composition. Therefore, it is difficult to compare the present results to those of previous studies that used different ceramics and etching protocols. For cementation of e-max CAD restorations, the manufacturer recommends an etching time of 20 s using a 4.9% HF gel. Both of water storage and thermocycling are used in vitro studies as a common method for testing dental materials to demonstrate their suitability for in vivo conditions, and among different storage conditions like distilled water, saline, 0.05% saturated solution of thymol, 0.5% chloramine-T, 2% gluteraldehyde, and 10% formalin solutions which were studied as storage media for bond strength tests (82, 83), we used the distilled water as storage media for 24 hr. before the test. De Munck, J in 2003 reported disitelled water storage is a common artificial aging technique in dental research (84).

Thermal cycling has been employed as a method to simulate clinical conditions. Mair et al reported that oral temperature ranged from −4°C-0°C when eating ice cream to 60°C-65°C when eating a hot cheese sandwich (85). Testing the samples by thermocycling speeds up the diffusion of water among changing the temperature produce stress at the interface of the two materials due to different coefficients of thermal expansion of both materials (86).

ISO TR 11450 standard (2003) recommended a short regimen of thermocycling is 500 cycle. Some previous studies calculated the number of cycles and reported 6000 thermal cycles are equal to 5 years of clinical use (87, 88). Therefore, 5000 cycles that is used in our study are equal to 4 years of clinical function. Celik et al compared the different methods of aging as thermocycling, water storage and mechanical fatigue. The thermocycling was the best method to test the quality of the bond, among all the aging methods. The bond strength was remarkably reduced after thermocycling (89). For this reason we used the thermocycling as aging method in our study.

Concerning mechanical cycling the amount of load exerted during mastication and swallowing varies from 70-150 N (90). Although, most of invitro studies used monotonic tests as compression, shear or tensile strength to examine the mechanical properties of the dental materials (91), the previous tests cann’t produce fatigue damage as that happened in the mouth. Therefore, the studies with fatigue tests should be done to get better clinical results (92). Oilo (93) classified the bond strength test into quantitative tests and qualitative tests. The quantitative tests predict the lifetime of the bond and the load capacity but qualitative tests study bond failures. Although, measurement of the bond strength can be done by clinical performance and laboratory methods (94).There is no laboratory test that will predict accurately the clinical performance of the dental materials (95). The laboratory tests can be dynamic or static test (96). In the static tests, force applied when the specimen is in stationery state while in dynamic tests force applied when the sample is in dynamic state. Shear bond test is static test. The static tests are classified into micro-tests where the bond area is <3mm^2 bond area and macro tests with >3mm^2 (97). The macro bond strength can be measured in tensile, shear or using a push-out protocol (98). In our study the bonded area was >3mm^2 so the test was macro bond strength test.

In the shear bond test (SBS) the two materials connected to each other by adhesive agent and loaded in shear until fracture happens. The nominal bond strength is calculated as the following maximum applied force / the bonded cross-sectional area (99). The shear bond test is considered as commonly used test (100). Some authors recommended a mandatory shear bond strength about 20 MPa for permanent success of restoration (101,102). ISO 10477 for polymer-based materials is recommended shear bond 5 Mpa (103). Regarding to the results of our test all the tested groups are fulfilled the ISO requirement of 5 MPa except group 4 (9.5 % Hydrofluoric acid etching for 90 seconds + Panavia SA plus cement) which was 3.89 Mpa.

In our study group 1 (sandblasting and multilink hybrid abutment) yielded the highest mean and standard deviation (SD) value of SBS (15.91±3.23MPa) and group 4 presented the lowest mean and SD value (3.89±0.59 MPa)**.**When we compared the mean SBS value of group 1 (15.91Mpa) with one previous study was done by Guilherme with 53.0 Mpa (mean) (78) .The difference in the results was clear because of additional procedures were done. The first procedure was using the Monobond plus universal primer in that study which contains MDP monomer responsible for chemical bond formation with oxide layer of titanium alloys which improve the bond strength and the second procedure was applying 5 % of Hydrofluoric acid gel (Ivoclar Vivadent) for etching lithium disilicate glass ceramics instead of 4.5 % for our study followed by ultrasonic cleaning and we did not because many studies shown that the presence or absence of hydrofluoric acid residue did not influence the bond strength between ceramic and resin (104) . Henrique et al (2014) (105) demonstrated that post hydrofluoric acid etching method may simply consist of rinsing with water for 90 seconds. From this point of view, our study support many previous studies which have shown that the bond of metal to composites can be enhanced by the use of metal primers (106,107,108). As chemical bonding of metal to composite resins involves coating the metal with primers that contain what are called functional monomers. These functional monomers create chemical adhesion between the resin restorative material and the metal. One study evaluates the difference in shear bonding strength between resin cements to dental materials when a universal primer (Monobond plus) was applied in place of a conventional primer and concluded that there were no significant differences in bonding strength depending on the type of primer used (109). According to Antoniadou et al (2000) (110), utilization of the Alloy Primer is simple, fast and effective to increase the durability and the bond strength between resins and sandblasted metallic alloys; however, this bonding depends on the composition of the alloy. In 2001, Yoshida, et al. (111) stated that the combined use of resin cements and an appropriate adhesive primer increases the clinical durability of restorations.

Comparing the mean shear bond strength values between all the groups in terms of the type of cement used, there was no statistically difference between them. That’s mean the two resin- cement systems used were not significantly different from one another when submitted to either to airborne-particle abrasion or than 9.5% hydrofluoric acid etching for 90 seconds. Both multilink hybrid abutment and Panavia SA plus cement gave higher bond strength with sandblasting and lower bond strength with 9.5% hydrofluoric acid etching for 90 seconds. Therefore, the cement type was not a significant influence on shear bond strength.

Concerning to the surface treatment type a wide range of mean shear bond strength values was noted between sandblasted groups (group 1= 15.91 Mpa, group 2 = 13.48 Mpa) and 9.5% hydrofluoric acid etched groups (group 3 =

5.35 Mpa, group 4 = 3.89 Mpa). Therefore, the results of our study indicated that sandblasting obtained higher bond strength values than 9.5% hydrofluoric acid etching for 90 seconds in both cementation procedures. This was likely due to the variation in the surface contact of testing methods, that is, airborne-particle abrasion and hydrofluoric acid gel etching coming into contact with the titanium alloy surfaces. During the HF etching process, the etchant was agitated and gas bubbles were formed. The complete contact of the etchants with the titanium alloy surface was likely compromised by the gas bubbles formation. It is though that stirring the etchant helped improve the contact area of chemical reaction. Therefore, the creation of gas bubble pockets during the surface etching of titanium alloy surface could also account for the increased variability of the range within tested groups. Many Studies have demonstrated that air abrasion can be an effective surface treatment to enhance the bond strength between resin composite and metal (112,113) and our study in agreement with them. Guilherme et al (78) evaluated the effect of different surface treatments of titanium alloy on bond strength of lithium disilicate ceramics and the results was reported that either airborne-particle abrasion or etching with 9.5% hydrofluoric acid for 30 seconds gave the highest bond strengths .However, increasing the etching time to 90 seconds reduced the bond strength significantly which is similar to our study results as increased etching time did not improve the bond strength, this likely as a result of over- etching or polishing the surfaces or the partial removal of the surface roughness leads to weakening of the surface structure bond .The results of the present study showed that shear bond strength between the titanium and lithium disilicate ceramics can be significantly affected by the etching time of titanium alloy which are not currently recommended by the manufacturers of the components.

The limitation of this in vitro study includes the low number of specimens tested and disc-shaped specimens were used instead of complete dental restorations. The medium used to perform thermal and mechanical cycling tests was distilled water instead of saliva in the oral cavity and they are chemically different .The bond strength of titanium alloy to lithium disilicate glass ceramics sensitive to chemical or mechanical influences in intraoral conditions. Another limitation is that no chewing simulator was used to simulate the dynamic forces in the mouth. Even we did thermocycling as aging process in our study which cannot simulate the ideal oral cavity condition. The study can be improved by using much more number of samples, metal alloys primer with different types of resin cement.

6. CONCLUSIONS

Within the limitation of this study we can conclude that:

1. The surface treatment procedure of titanium alloy has a significant influence on the shear bond strength between titanium and lithium disilicate glass-ceramic.
2. The cement type was not a significant influence on shear bond strength.
3. Regardless of the type of cement used the sandblasting is an effective method to increase bond strength because it improved the bond strength compared to 9.5% HF acid for 90seconds.
4. Increasing the etching time of 9.5% Hydrofluoric acid did not improve the results.

Therefore, the null hypothesis of this study, that different surface treatment and cementation procedure combinations will not affect the shear bond strength values at the titanium alloy to lithium disilicate ceramic interfaces was rejected.

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