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THE EFFECT OF DIFFERENT METAL PRIMERS ON SHEAR BOND STRENGTH BETWEEN HEAT CURE ACRYLIC RESIN AND DIFFERENT METAL ALLOYS AFTER THERMOCYCLING

MASTER THESIS

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ABBREVIATION AND SYMBOLS

RPD: Removable Partial Denture.

CP Ti: Commercial Pure Titanium.

Co-Cr alloy: Cobalt-Chromium alloy.

Ni-Cr alloy: Nickel-Chromium alloy.

PMMA: Poly methyl methacrylate.

4-META: 4-methacryloyethyl trimellitate anhydride.

MDP: 10-methacryloxydecyl dihydrogen phosphate.

VBATDT: 6-4-Vinylbenzyl-n-propyl amino-1, 3, 5-triazine 2, 4-dithone.

BPDM: Carboxylate monomer.

Au: gold.

Ir: iridium.

Pt: platinum.

Pd: palladium.

Ag: silver.

Cr: Chrome.

Mo: molybdenum.

MPa: megapascal.

GPa: gigapascal.

ISO: International Organization for Standardization.

Psi: Pounds per Square Inch.

EDS: Energy-dispersive X-ray spectroscopy.

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

µm: micrometer unite.

SBS: Shear bond strength.

N: Newton unite of force.

ANOVA: Analysis of variance.

Tukey HSD: Tukey method, honest significant difference test.

P: P Value (Calculated Probability).

et al: and others.

MMA: Methyl methacrylate.

ABSTRACT

Purpose: The purpose of this in vitro study was to evaluate the effect of metal primers on the shear bond strength (SBS) between Cobalt-Chromium alloy, Nickel-Chromium alloy, Titanium alloy and heat cure acrylic resin after thermocycling.

Material and Methods: Total number of60disc-shaped wax patterns (10mm in diameter and 3mm in thickness) were casted in Cobalt-Chromium, Titanium ($Ti_{90}Al_6V_4$) Nickel-Chromium. After casting, the discs surface was sandblasted with110μm Aluminum oxide for 5 seconds. Specimens of each metal were divided into two groups (n=20) and received one of the following metal primers: (1) Alloy Primer (Kuraray), (2) Z-Prime Plus (Bisco). The specimens were divided into 6 subgroups (n=10) according to the metal primer used. Group1: Cobalt-Chromium / Alloy Primer, Group 2: Cobalt-Chromium / Z-Prime Plus, Group 3: Titanium / Alloy Primer, Group 4: Titanium / Z-Prime Plus, Group 5: Nickel-Chromium / Alloy Primer, Group 6: Nickel-Chromium / Z-Prime Plus. All the specimens were stored in the distilled water at 37℃ for 24 hours after polymerization we did thermocycle (5000 cycles at 5℃-55℃ with a 30- second dwell time for each temperature). After thermocycling, the specimens were tested in a universal testing machine at a crosshead speed of 0.5mm/min in shear mode. Data (MPa) were analyzed using one-way ANOVA and post Hoc Tukey ($P < 0.05$).

Result: The one-way ANOVA indicated that SBS values varied according to type of metal and metal primer used ($P < 0.05$). The highest SBS was recorded for Group 4: Titanium / Z-Prime Plus (16.93±3.3) MPa. The lowest SBS was recorded for Group 5: Nickel-Chromium / Alloy Primer (1.20±0.22).

According to the metal type the highest SBS was for Titanium alloy (13, 18±4, 59) MPa. And for the metal primer the shear bond strength of the Z- Prime Plus groups (9.81±6.75) MPa was statistically significant higher than the shear bond strength of Alloy primer groups (p=0.0001).

Conclusion: It could be concluded that using Z- Prime Plus after sandblasting significantly improves the bonding of heat cured acrylic denture base resin to Titanium alloy.

Keywords: shear bond strength, titanium alloy, thermocycling, metal primer.

1. INTRODUCTION AND AIM

Removable Partial Denture (RPD) is one of the most used treatment modality in the partially edentulous patient(1).RPD Are mainly fabricated with a metal framework which is commonly constructed from the base metal alloys such as commercially pure Titanium (CP Ti) or Titanium alloy, Cobalt-Chromium (Co-Cr alloy)or Nickel-Chromium (Ni-Cr alloy)and heat cure acrylic resin material which is based on the Poly methyl methacrylate (PMMA), and a monomer as denture base material.(2)The differences in the thermal expansion between acrylic resin and metal alloys framework and the polymerization shrinkages of Polymethyl methacrylate acrylic resin during laboratory procedures may result in the bond failure between the heat cure acrylic resin and metal.(3)To enhance this kind of treatment it has to improve the overall quality of the bonding between acrylic resin and metal framework.(3)Generally during the construction of the metal framework we have to incorporate some elements that provide mechanical retention between the denture base acrylic resin and metal alloy structure such as loops, beads, bars and preformed mesh or using electrochemical etching, silica coating, metal primers, and sandblasting to improve the bonding between these materials.(4,1)Although the utilization of the mechanical retentive system may result in a bad adhesion and micro leakage of oral fluids, causing problems such as discoloration, foul smell and increase the susceptibility of the prosthesis to fracture.(5)These problems are solved, after using of chemical etching. But the chemical etching is limited to the Nickel-Chromium alloys. According to several studies, the chemical bonding had a superior bond to the mechanical retention systems. (6, 4)

Currently, the bonding between the heat cure acrylic resin and metal alloy framework has been improved by using different types of metal primers. That contain different functional groups like 10-methacryloxydecyl dihydrogen

Phosphate (MDP) or 4-methacryloyloxyethyl trimellitate anhydride (4-META).[2]Other metal primers are available.

The bond strength between the heat cure acrylic resin and the metal framework does not only depends on using of metal primers. But also on metal primer composition, type of acrylic resin used and composition of the metal alloys. [1, 2]

Dewan et al. [7] refined the metal framework by laser etching and applying of metal primers to the base metal alloys to improve the bond of resin to the non-precious castings. The highest bond strength founded in the samples that treated with both laser etching and metal primers.

The aim of our study was to evaluate the effect of different types of metal primers that contain different functional groups such as Alloy Primer-Kuraray contains 6-4-Vinylbenzyl-n-propyl amino-1, 3, 5-triazine 2, 4-dithone (VBATDT) and 10-methacryloxydecyl dihydrogen phosphate (MDP). That enhances the bond strength to precious and non-precious alloys. And Z-Prime plus Bisco that has two active monomers (MDP) a phosphate monomer, and (BPDM) Carboxylate monomer on shear bond strength between heat cure acrylic resin and different metal alloys as Cobalt-Chromium alloys, Nickel-Chromium and Titanium alloys after thermocycling.

2. REVIEW OF LITERATURE

It has been found that the causes that make patient to be edentulous not only dental diseases such as dental caries or periodontal problems. But also there are other factors as patient attitude, behavior and general health problems. That lead to tooth loss then the patient will be treated by one type of prosthodontics treatment options. Consequences of tooth loss are aesthetics issues, phonetic problems, drifting, tilting or over eruption of teeth, loss of masticatory efficiency, loss of vertical dimension and loss of alveolar bone. [8, 9]

Prosthodontics is a branch of dentistry that deals with replacement of teeth, soft or hard tissues or other jaw structures by artificial devices, to restore and maintain the oral function, comfort, appearance and health of the patient.[10]

2.1. There are four branches of Prosthodontics

- Fixed prosthodontics.
- Removable prosthodontics.

  1-Compelet Dentures.

  2-Removable Partial Dentures.
- Maxillofacial prosthodontics.
- Implant prosthodontics.[10]

2.1.1. Fixed prosthodontics

It is a science that is dealing with replacement and restoring missing teeth by artificial substitutes that are attached to natural teeth, roots or dental implants the patient cannot remove these substitutes from the mouth by them self. [11, 10]The main goals of this kind of treatment are to restore: function, aesthetic, and comfort.[11] So it can convert the unhealthy and compromised occlusion into normal comfortable occlusion.[11]Fixed prosthodontic treatment indicated when one or two adjacent teeth are missing in the same arch with present of suitable abutment teeth and healthy supporting tissues. It can range from one tooth restored by cast crown or cover more than one single tooth with bridge or to more complicated option as implant-supported restorations.[11] Fixed Prosthodontics contraindicated when the supported structures are: missing or diseased or present of unsuitable abutment teeth, general patient health is not good or poor oral hygiene or patient has poor oral habits and economic problems.[11]

2.1. 2.Types of fixed prosthodontic restorations

Fixed restorations can classify as following.[10, 11]

1-Depending on the type of connector	2-Type of material used	3-Duration of use	4-Type of abutment:	5- Length of the span:

1-Fixed fixed partial denture. 2-Fixed removable partial denture. 3-Removable fixed partial denture.	1-All metal crowns. 2-Metal ceramic crowns. 3-All acrylic crowns. 4-Ceramic veneer. 5-Acrylic veneer.	1-Permenet fixed partial dentures. 2-Long span bridges [Interim, Periodontally weak abutment, Splints].	1-Normal abutment. 2-Cantilever. 3-Pier abutment. 4-Mesially tilted abutment.5-Endodontically treated abutment.	1-Long span bridges. 2-Short span bridges.

Table (1) classification of fixed prosthodontic restoration.

2.2. Removable prosthodontics

Removable Prosthodontic treatment defined as the replacement of the natural teeth in the arch and their associated structures by artificial substitutes. Removable prosthodontics can classify as

1- Complete denture.
2- Removal partial denture.[16]

The denture is an artificial replacement appliance for missing natural teeth and supporting structures. The replacement of missing teeth enhances the patient aesthetically by replacement lost facial contours, vertical dimension. And restores masticatory efficiency, phonetic and tissue preservation. [16]

2.2. 1. Complete denture

A complete denture is a full replacement of the natural teeth and their associated structures with artificial substitutes. [10, 16] Complete denture indicated for the completely edentulous patient or partially edentulous patient when the remaining teeth cannot support a removable partial denture or cannot save it or when no acceptable alternatives are available, or the patient refuses alternative treatment. And contraindicated in some cases as physical or mental illness affects the patient's ability to cooperate during the fabrication of the denture and to accept or wear the denture, or a patient has hypersensitive to denture materials. Complete denture may be constructed from metal or acrylic resin since the mid of 1940's the complete denture delivered to the patient in the form of acrylic resin poly methyl methacrylate known as PMMA this material performed by polymerization. [10, 6]

2.2.2. Removable partial denture

Removable partial denture restores oral functions, comfort, appearance, and health of the patient by restoring natural teeth and replacing missing teeth and craniofacial tissues with artificial substitutes. The removable partial denture is prosthesis that replaces some teeth in the partial dentate arch and can remove from the mouth and replaced again by the patient. [12,13]It can classify as

1- Removable complete denture prosthodontics.
2- Removable partial denture prosthodontics. [10]

The loss of teeth will effect on the ridge volume both height and width, as the change in the anatomy has reported to a variable across patient groups. In general, the bone loss in the posterior more than anterior and the mandibular ridge more than the maxillary ridge, as a result of this normal philological process the mandibular arch becomes wider than maxillary arch which becomes constricted, and the fabrication of the dental prostheses becomes more challenged due to this anatomical changes.[12]

2.2.2.1. The classification of partial edentulous arches according to Kennedy method of classification.[12]

Class 1- Bilateral edentulous areas located posterior to the natural teeth.

Class 2- A unilateral edentulous area located posterior to natural teeth.

Class 3- A unilateral edentulous area with natural teeth remaining both anterior and posterior to it.

Class 4- A single, but bilateral crossing the midline, edentulous area located anterior to the remaining natural teeth. [12, 13] In some cases, there is one of these classifications with another edentulous are which defined as a modification.

The removable partial denture is easily repaired and adjusted to functional restoring when there is no distal teeth are a present or long span of lost dentition, supports periodontally involved teeth. It is suitable for these cases of several missing teeth in the same quadrant or both quadrants of the same arch, for replacement of missing teeth when the fixed or implants treatment cannot do it, temporary replacement for missing teeth in a child or as a splint to support periodontical involved teeth. Removable partial denture contraindicated when there is a lack of suitable teeth in the arch to retain, support and stabilize the removable partial denture. Rampant caries or severe periodontal problem that threaten the remaining teeth in the arch.[12, 13]

2.2.2.2. The components of removable partial denture

The removable partial denture parts are the major connector which is a part of RPD that connects the other parts of the prosthesis located on one side of the arch with those on the opposite side this component provides the cross arch stability to prevent denture displacement by functional stresses.

The minor connector connects the major connector or denture base with other parts of removal partial denture, such as clasp assembly, indirect retainer, and sometimes the minor connector continuous with some other part of the denture. [12, 13]The direct retainer is a clasp or attachments applied to an abutment tooth for the purpose of holding the removable partial denture in position. Indirect retainer if the prosthesis has distal extension base it is a part of the removable partial denture that prevents rotation of denture during the function.[13]Framework is the cast metal skeleton that provides support for remaining components of the prosthesis. Artificial teeth constructed from either acrylic or porcelain. Stabilizing or reciprocal components as a part of a clasp assembly.[12]Rest it is a component of the partial denture that placed on an abutment to prevent the movement of the denture in the gingival direction and transmits functional forces to the tooth. Rest can be classified according to the position there are occlusal, lingual or incisal rest.[13]

2.2.2.3. Removable partial denture construction

The materials that used for removable partial denture construction are metal alloys, acrylic resin. Metal is an element which ionizes positively in solution. Alloy is the combination of two or more metals and contains the best properties of many metals for specific purposes. The microstructure of metal alloy is grains crystals – regularity and repetition crystalline and grain boundaries no regularity and no repetition amorphous. Solid solution alloy is a combination of two or more metals. Which are completely soluble in each other in both liquid and solid state. It is stronger, harder but less ductile than the constituent metals due to the difference in atomic size crystalline structure of the alloy less than 15% solution hardening. It is more ductile and resistant to tarnish and corrosion than other types of alloys as solid solution alloy. The alloys have been used in dentistry for many years ago to replace missing teeth, the first type of alloy used was gold because it was easy to purify, melt and manipulate. Other metals that used for dental treatment as platinum and most of the pure metals lack some properties to use for an extensive restoration. For this reason, it is better to use alloys which are mixed with metal and nonmetals together to get appropriate chemical and physical properties. (14)

2.3 .Maxillofacial Prosthodontics

The maxillofacial prosthodontics is the branch of prosthodontics that replace or restore the congenital or acquired defects in the head and neck regions of the patient by artificial substitutes, and these substitutes range from minor functional disabilities to major with or without aesthetic deformity. Craniofacial deformities may result from trauma or cancer or congenital defect as cleft lip or cleft palate. (10, 31)

2.4. Implants prosthodontics

Implant prosthesis is the graft set firmly or deeply into or onto the alveolar process that may prepare for its insertion. The dental implant is a substance that is placed into the jaw to support a crown or fixed or removable denture.(10, 11)

Dental implants indicated for single tooth replacement or for completely edentulous patients with severe bone resorption and partially edentulous patients with weak abutments. Advantages of dental implants are bone preservation, increase masticatory efficiency, provide more natural appearance than other prosthetics replacement and more comfortable for the patient. Disadvantages of using dental implants are very expensive, contraindicated for the medically compromised patient, long duration procedure and need more patient cooperation and they cannot universally place due to anatomical variations. (10, 11)

2.4.1. Dental implants classification

Depending on the placement within the tissues implants can be classified as follows: Epiosteal implants, Transosteal implants, Endosteal implants, Root form implants, Plate form implants. Depending on the materials used metallic implants titanium, titanium alloy, cobalt chromium alloy and non-metallic implants ceramics, carbon. [11] And according to the reaction between implants and bone

1-bioactive (hydroxyapatite).

2- bio-inert implants (metals).[10]

2.3. Classification of casting dental alloys

General classification of metal alloys are

1-The noble metals which have good resistance to corrosion and tarnish during laboratory work for example gold [Au] platinum group thatmay be contained heavy group as iridium (Ir) or platinum (Pt) or light group as palladium (Pd). Noble metal alloys presented in high noble alloys form in which the gold is more than 40%. And the other 60% other noble metals. The amount of noble alloys is 25% and the content is gold. And predominantly base metal alloy form the base metal alloys form about 75% and more and noble metal alloy less than 25%.[14]

2-Precious metals noble metals- silver (Ag).[16]In 1970 the base metal alloy became widely used in dentistry which can define as a subrogation of solid solution alloys that do not contain any noble metals, and it used instead of gold alloys type Ⅲ and Ⅳ in some cases. The base metal alloys used for partial denture framework construction, crown, bridge, the base of complete denture and for dental implants.[16]

2.2.1. Types of base metal alloys

There are three types of base metal alloys.

1-Nickel-Chromium.

2-Cobalt-Chromium.

3-Titanium alloys. (14)

Property	Cobalt-chromium	Nickel-chromium
Density[g\cm3]	400	350
Vickers hardness	850	600
Tensile strength[MPa]	≤ 1500	≤ 1350
Proportional limit[MPa]	700	500
Casting shrinkage[%]	2.5	2.0
Modulus of elasticity[GPa]	220	185
Elongation[%]	1.5	2.5

Table (1) Properties of Co-Cr and Ni- Cr alloys. (16)

Grains and crystal structure of alloys

Crystal structure of dental alloys is similar to the crystal structure of salt or ice. The grains that formed after freezing of the molten alloy as it called in the metallurgy clearly can be seen under a microscope. These crystal increase in size slowly and go into each other in same behavior of water crystals. [14]Since 1928 the Cobalt-Chromium alloys used for partial denture fabrication and then the Nickel-Chromium also used. These type of base metal alloys became the predominantly option for partial denture framework fabrication. [15]The Cobalt-Chromium alloys mainly used for partial denture casting due to it has high yield strength and elastic modulus. The majority of Ni-Cr alloys used for crown and other small casting prostheses.[16]

The base metal alloys have a physical proprieties such as high strength and corrosion resistance make them suitable for a dental application. Although the Ni and Co are the main elements for these base metal alloys, there are other elements as (Cr, and Mo), and also may have one of these metals elements beryllium, iron, nitrogen, aluminum, titanium, manganese, silicon and zirconium, these elements added to improve the mechanical properties. Adding of silicon and manganese enhance the castability of the alloy. Aluminum increases the yield and tensile strength. [15] The pure form of base metal alloys cannot use in the dental restoration due to the high susceptibility of the pure form to be corroded in the oral environment. Except for titanium which can utilize in the nearly pure form to make dental implants. For that, the base metals should be in alloys form to provide the strength, wear resistance and flexibility that necessary for restoration. [14]

2.2.2. Properties of Co-Cr alloys for RPD framework production

According to the requirements of ISO 6871-1:1994 for base metal alloys to be used in casting partial denture fabrication the total amount of the Co, Cr, and Ni should not be less than 85% of weight, and for Mo and Cr should not be less than 4%, 25 %. (32)Cr is the primary element which is added to improve alloy strength through carbide formation (thin an oxide layer) to increase the resistance to oxidation and corrosion, although the good effects of this element on the electromechanical and mechanical proprieties, the over addition of this element may produce Cr-rich sigma phase, which decreases the resistance to corrosion and make the prostheses hard and brittle.Ni-Cr based alloys provide less corrosion and tarnish resistance, strength, modulus of elasticity and biocompatibility than Co-Cr based alloys. (33) The Ni-Cr-Mo based alloys exhibit corrosion resistance from 0.65 to 3261 g/cm2 although carbide formation is the primary strengthening mechanism. (32)

2.2.3. Desirable Properties of Dental Casting Alloys

1-Tarnish and Corrosion resistance

Tarnish is a thin layer of a surface deposit that is attached to the metal surface, and corrosion is one of the physical dissolutions of dental material in the oral cavity, and there some noble metals that do not change in the oral environment as palladium and gold so they have more corrosion resistance, and some elements that form thin surface film, which prevent any subsurface reaction as titanium and chromium. (15)

2-Biocompatibility

All the metal and metal alloys that used for dental restoration must be tolerated in the oral environment and do not produce any toxic or harmful products. (15)

3-Thermal properties

The melting point of casting alloys should be suitable small enough to create smooth surfaces when it becomes in contact with the mold wall which finally provides well fit casting prostheses. (15)

4-Strength

Dental casting alloys must have adequate strength especially when they used for long-span frameworks. (15)

Titanium and titanium alloys

Commercially pure titanium alloys and Titanium alloys have occasionally been used to make removable denture frameworks because of excellent properties such as corrosion resistance, biocompatibility and mechanical properties, the rigidity of low elastic modulus of connector can be improved by increasing its thickness or changing its design, increasing the rigidity has another beneficial effect by reducing deboning between resin and the metal framework, and the lower yield strength and tensile strength and higher percent elongation of CP Ti makes the adjustment of the cast clasps more easily.(15)There are some characteristics of commercially pure titanium and it's alloys that limiting its use and increase difficulties in the CP Ti casting process such as low density. The low density and porosities of CP Ti make it easy to identify by conventional radiography, high melting temperature and reactivity at high temperature. Despite these make the laboratory casting of CP Ti more challenge, since the internal porosity within the clasp can lead to clasp fracture, and the reaction layer on the surface must be removed chemically with hydrofluorosilicic acid or by grit blasting and rotary instruments. Due to the high rate of oxidation which is more than 900 ℃ and high melting point (1668℃) make them require a particular casting machine with the arc-melting capability and an argon atmosphere. (15) There are new casting machines for CP Ti, the high electrostatic binding capacity of the titanium surface oxides results in higher plaque adherence to titanium frameworks compared with other base metal frameworks. And some technological problems remains such as porosity and inadequate mold filling, it should control the framework quality since voids and pores may affect fatigue resistance of RPD framework that subjected to repeated load cycles during chewing and frequent insertion and removal from the mouth. (17)

2.2.4. Biological hazards of base metal alloys

Allergy-inducing substances can be dissolved to the oral environment from an alloy or spread to the oral cavity by the airborne particulates during grinding and polishing and propagated by vapor during casting, the amount of vapor released during casting depends on the vapor pressure the quantity of the cast mass, and the atomic concentration of the element.(15)

2.3. Acrylic Resin

Polymethyl methacrylate is the material of choice for denture base construction because of its properties such as approbatively working time, easily processing, good aesthetic, economic and accurate fit. Despite these excellent properties, there is some enhancement for fracture resistance in this material by adding glass or aramid fibers especially in the distal extension partial denture or temporary fixed partial denture because they are more subjected to fracture.(18) Polymerization reactions the formation of the polymethyl methacrylate by addition polymerization of methyl methacrylate through mixing the monomer methyl methacrylate with powder partially polymerized methyl methacrylate after mixing the polymer powder with monomer liquid the dough stage formed which is packed into the flask, after setting of teeth, then cured to form denture.(18)

2.3.1. Types of acrylic resin

1-high- impact resins: specially formulated acrylics or elastomers as butadiene-styrene rubber spheres from 5 -10% added to the powder to enhance the impact resistance of denture, color stability and improve toughness as well as increase fracture resistance in compare to the conventional one, but there is no special processing for curing.[19]

2- Pour- type resins: the resin powder of liquid is pour-type resin contains small particles that when mixed with liquid, produces a fluid mix. The mixed material must be rapidly pour or injected into a hydrocolloid mold and polymerized under pressure 20-30 psi in 106 °F\ 41°C water for about 15 min these resins may be weaker than conventional one due to the residual monomer content and become more susceptible to distortion than the other one. But new techniques and materials have eliminated many of these problems; it can be injection molded using customized processing units, the overall time for processing is shorter than the traditional acrylic resin, and improvements in the appearance and properties of this resin denture continuously made. Also, can use this material for construction of RPD or for repairing of the fractured complete denture.[19]

3-rapid-cure resins: are hybrid polymers that incorporate both chemical and heat-activated initiator and that cure rapidly in boiling water; it is weaker than conventional acrylic resins.[19]

4-light- cure resins: it becomes more popular because of speed and convenience, usually based on (urethane ollgomers) and some comprise a urethane dimethacrylate acrylic copolymer with microfine silica filler particles. Also they contain a photoinitiator responding to blue light (400-500nm) and in particular processing unit, and they have many advantages than conventional one such as good mechanical properties especially in the new systems, but the major advantage of light- cure systems is that they eliminate the need for the loss of wax technique nor require the boiling out or the investing .also the esthetics of this urethane- based is good because it is stain and plaque resistance equal to or superior to the acrylic resin, these materials used for provisional restorations, RPDs and night guard. [19]

The most common acrylic resin used for partial denture construction is heat cured acrylic resin (PMMA). And it has been found that the best time for heat cure acrylic resin to get high impact strength is 70c for 9 hours. [20]

2.3.2. Physical and biological considerations of conventional heat-cured denture bases

Methacrylate resin PMMA dentures have low thermal conductivity, linear expansion after undergoing to water absorption by diffusion which offsets polymerization shrinkage, low abrasion resistance the acrylic teeth and denture base may undergo to wear if the patient uses abrasive agents or stiff brush to clean it, also the resin is soluble in organic solvents upon exposure to acetone or alcohol, the polymer network swells as the resin dissolves, leading to irreversible damage to resin surface and biocompatibility of denture base methacrylate resin to the surrounding oral environment. However, water sorption, cracks, surface imperfections, and microporosity of denture base makes the attachment of microorganisms most common especially in absent of adequate oral hygiene such as candida-Albicans which cause some pathological lesion as denture-induced stomatitis.[8]

2.3.3. The bonding of acrylic resin to metal alloys

The traditional method for denture base resin to be attached to metal framework by mechanical retentive elements such as beads, mesh, loops and undercut as mechanical ways for retention, or adding elements as external or internal finish lines to ensure that there is a sufficient bulk of acryl for strength ,but this macro-mechanical may affect the acrylic matrix because it will be stress concentration area, although there are other methods as sandblasting, silica coating, chemical etching, electrolytic etching and using of metal primers.[21]

2.4. Sandblasting

Sandblasting is one of pretreatment technique to metal surface to increase the bonding of acrylic resin to metal alloys. In this technique the morphology and the composition of metal alloy will be changed but this changes will not have any passive effect on the fitting of restoration, and the noble alloy will be affected more than base metal alloy.(22) For example the alumina particles that embedded in the alloys after sandblasting will increase the content of alumina content from 14%to 37% as measured by Energy-dispersive X-ray spectroscopy (EDS), also using tribochemical silica coating leaves a small layer of silica particles on the surface which increase the content of silica from 12% to 20%, after ultrasonic cleaning of tribochemical silica-coated alloys and sandblasted alloys the resin bonding may increase due to removal of loose particles of silica or aluminum without any effect of composition. (22)

2.5. Metal primers

First generation metal adhesion system was in 1978 Takeyama et al when he increase the bond strength of acrylic resin to 175 kg\cm with bovine enamel by 4-methacryloyloxethyl trimellitate anhydride which is a newly discovered materials, after mixing 4-META powder with water. The 4-MET will be formed through rapid hydrolysis reaction, and when mixed with solvent such as ethanol esterification will happen in which one of the carboxylic groups may react with subsequent inactivation of the carboxylic group for demineralization and adhesion promotion as meta-dent used for heat cure acrylic resin, it contains the conventional polymethyl methacrylate acrylic resin and the monomer contains 5% of 4-META.(23) In 1985 Meta-fast was introduced to the UK as autopolymerizing denture base acrylic resin with composition similar to acrylic solder 93% methyl methacrylate and 7% 4-META, and other examples as Panavia cement, Epricord Opaque Primer(MDP),Clearfil SE Bond primer, etc.(23).

The second generation of metal adhesion system in 1980s Kojima synthesized 6-(4-vinyl benzyl-n-propyl) amino-1, 3, 5-triazine-2, 4-dithiol, VBATDT as a first adhesive monomer for noble alloys. In 1994 V-Primer was the first marketed Thiol group containing metal primer and used for noble metal alloy then Alloy primer for noble and non-noble metal alloys, there are some reports that the polymerization reaction of acrylic resin affected by the VBATDT monomer that contains benzoyl peroxide-amine initiator system, as Panavia. Adhesive monomer products that contain sulfur component Tokuyama dental Corp., Tokyo, Japan. Metaltite is an adhesive monomer that contains Thiouracil group, used for noble metal alloys, M.L.Primer that contain Disulfide group used for noble and non-noble metal alloy. Metal primerII contains thiophosphate group and used for noble and non-noble alloy. [23]

2.5. Ultimate strength

The stress at which fracture occurs is called the ultimate strength. If the fracture happens from tensile stress the property is called the tensile strength, but if it happens in compression the prosperity called compressive strength, and if it happens in shear the strength will be a shear strength. Usually, the bond between two materials measured under tension or shear strength to test the amount of this bond, and this bond may be chemical or mechanical or a combination of two of them. [14, 15]

2.7. Measurements of adhesion bond strength for dental materials

There are some laboratory methods to test the adhesion bond strength of restorative materials such as shear bond strength test, tensile bond strength test, and flexural bond strength test.

2.7.1. Tensile bond strength test

In the metal-ceramic case the test consists of porcelain bonded to one end of a metal rod or between two ends of rods when the tensile longitudinal force applied along the long axis of the rod to separate the porcelain from metal.(24) This type of test can measure by dividing the maximum force at which the failure occurs by the cross-sectional bonded area. The result of tensile bond strength test affected by the geometry of the specimens and the occurrence of non-uniform distribution stress of force during the load application.(25)Although the measurement of tensile bond strength alone it does not give all the information about the bond strength of materials, it should measure the cohesive failure of porcelain.(24)

2.7.2. Shear bond strength test

Shear bond strength test is a test in which two materials are connected to each other by using adhesion agent under force until the failure occurs.(26)It measured by dividing the maximum applied force that responsible for the separation of the bond by the bonded cross- sectional area.(32)Advantages of shear bond strength test it has clear test protocol, simplicity of use, and the test result rapid appeared.(27) But this test needs large size of the specimen, which increases structural flaws of ceramic materials that will lead to premature failure of the specimen before reaching the maximum level of the force that responsible for the separation of the bond.[28]

2.7.3. Flexural bond strength test

Flexure or bending tests widely used in ceramics industry due to the problems of brittle materials when loaded under direct tension. This type of test measured in a three-point flexure test or a four-point flexure test, in both cases the applying force increased until the separation occurs. [29]

2.7.3.1. The three-Point bending test

The three-Point bending test is a simple, reliable and sensitive way to test the dental ceramics strength. [35] According to the international standards organization (DIN ISO 9693: 19999) that supports to use the three-point test as Schwickerath crack initiation test to measure the bond strength between porcelain and metal alloys.[36]In 2011 measure the bond strength of veneering ceramics to zirconia cores by using Schwickerath crack initiation test by Kosyfaki.[30]

2.7.3.2. The four-point bending test

It is used to measure the adhesion at a biomaterial surface. [37]This test can determine the interfacial fracture toughness of several types of metal-ceramic bonding systems regarding of strain-energy release rate. [38, 39, 40]In 1989 this test was first introduced by Charalambides et.al.[31]However, there were some problems in the specimen preparation and pre-crack producing. [41]

3. MATERIAL AND METHODS

In this study we used 60 disc-shaped metal alloy which were 3 mm in thickness and 10 mm in diameter. 20 discs were produced from Nickel-Chromium 20 from Cobalt-Chromium and 20 disks from Titanium alloy according to manufacturer instructors.

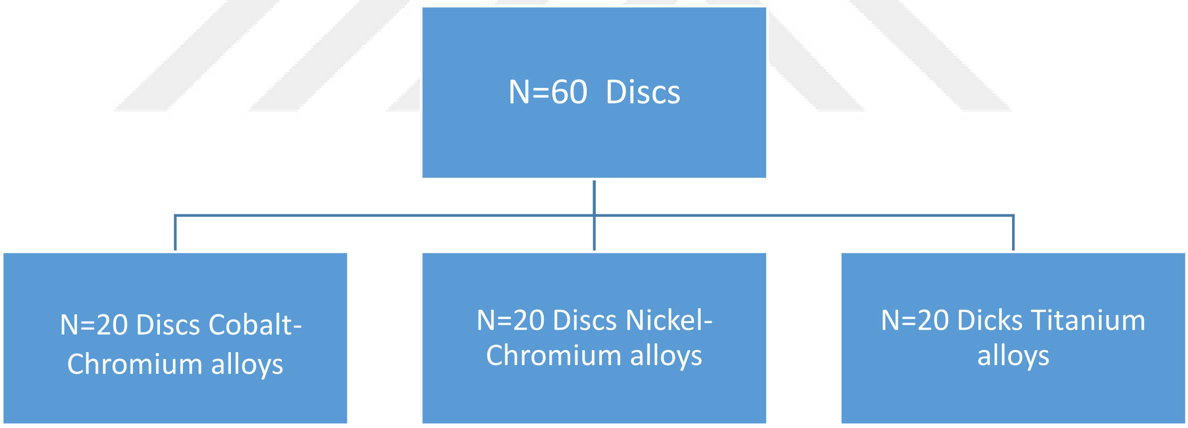


Figure (1) flow-chart illustrate the number and the materials used in the test.

3.1. Fabrication of metal alloys disks

We used the CAD-CAM system to fabricate wax discs from wax blocks [Kronenwachs, Bego Germany] we put the sprue attached in a silicon crucible former and invested by using phosphate bonded investment material(Bellasun, Bego Germany). Then the casting was done in the induction casting machine (Imes, Germany). Every disc has particular dimensions 10mm in diameter and 3 mm thickness. The 20 Cobalt- Chromium metals discs were fabricated from Metaplus UNI, Eleksan, Germany and 20 Nickel-Chromium discs were fabricated from SOLERA VK, Germany and 20 Titanium alloy discs were casted from Dent İndex, Turkey as manufacturer instructions by a magnesia-based investment in an argon arc-centrifugal casting machine.

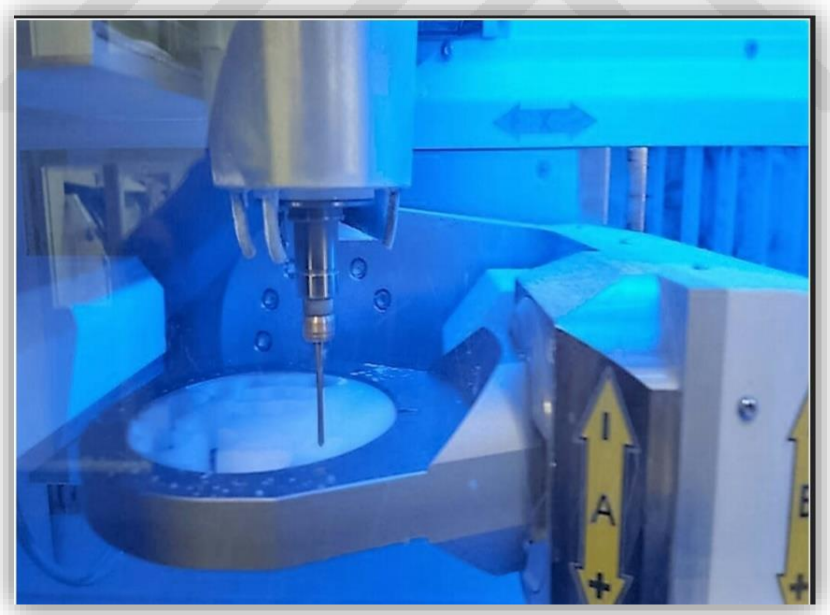


Figure (2) cutting of wax by CAD-CAM system

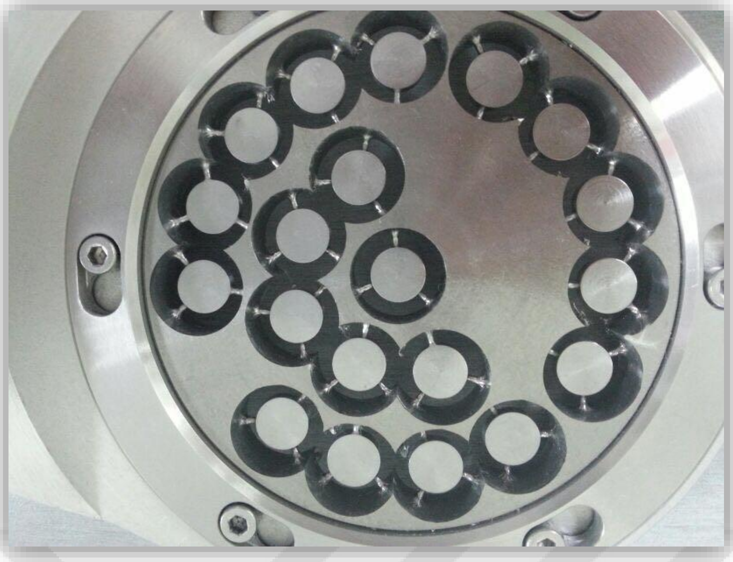


Figure (3) for metal disks

Figure (4) the sprue attached to the wax disk

Figure (5) all the attached sprue wax disks connected to crucible former.

Figure (6) Vacuret-delta machine used to mix phosphate bonded investment material for metal casting.

Figure (7) Imes, metal induction casting machine

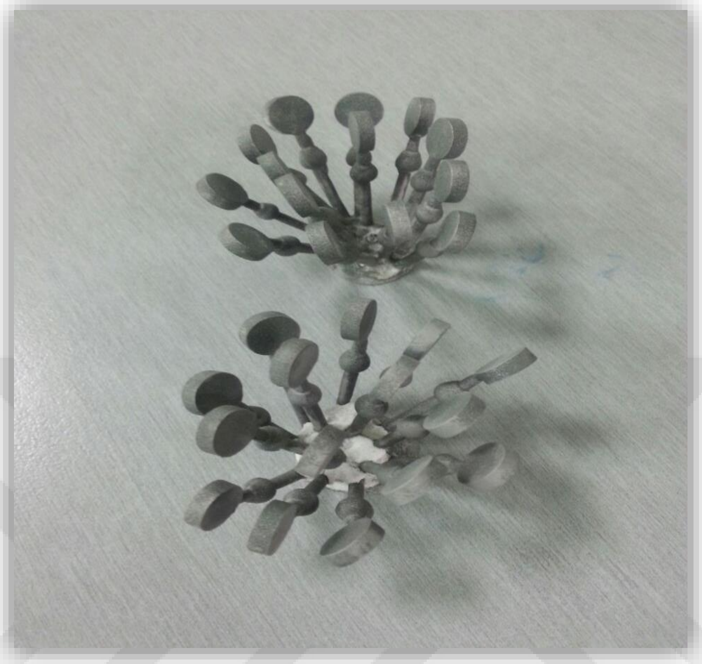
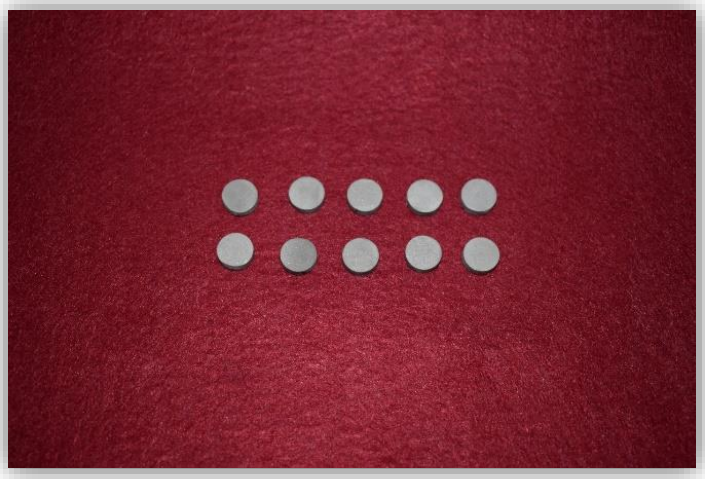


Figure (8) metal disks after casting



Figures (9) metal discs after finishing

Brand	Manufacturer	ISO Number
Cobalt-Chromium alloy	ELEKSAN Metaplus UNI , Germany	DIN 13912,EN ISO 6871
Nickel-Chromium alloy	SOLERA VK, Germany	EN ISO 6871-1, EN ISO 6871-2
Titanium alloy	Dent İndex, TURKEY	TS EN ISO 22674 Standards ISO 9693TYP5
Alloy-primer	Kuraray, JAPAN	
Z-Prime Plus	BISCO,Inc. 1100 W. Irving Park Rd. Schaumburg,IL60193 U.S.A 847-534-6000 1-800-247-3368	
Heat -Cure Acrylic Resin	Heraeus Kulzer GmbH GrunerWeg 11 63450 Hanau, Germany	ISO 10993 and ISO 7405
Self -Cure Acrylic Resin	IMICRYL,TURKEY	ISO 13485:2003

Table (2) materials used in the test.

3.1.2 Surface treatment

Metal disc's surface were subjected to Aluminum oxide. The particles size were 110 μm and sandblasted for 5 sec at 80 psi, at 45- degree angle. There was 5mm distance from the metal surface and the nozzle of the machine (Star Dental, TURKEY).

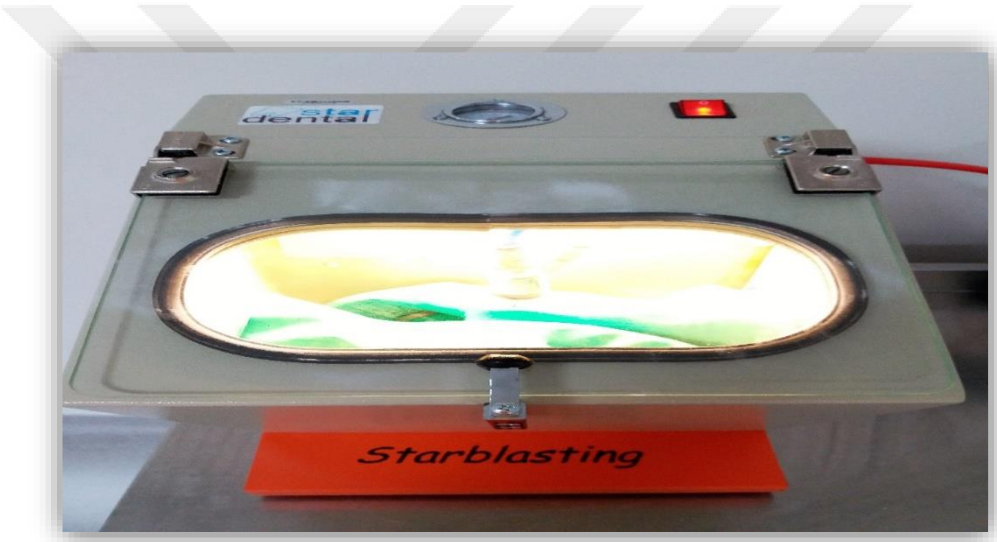


Figure (10) Star dental machine for sandblasting.

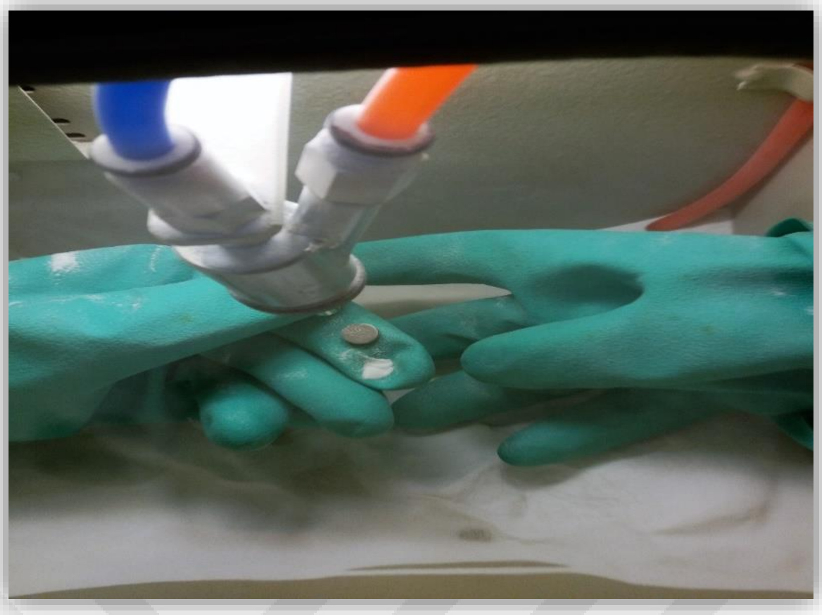


Figure (11) during sandblasting

Figure (12) the final shape of metal discs after sandblasting.

After we finish the sandblasting of all discs, we used silicon ring with inner diameter 7mm and 2.5 mm in height to make a wax disc on the sandblasted metal surface by dropping technics. We used baseplate wax (Cere Wax, Turkey) for this step.

Figure (13) the samples after adding wax disks.

Inside all the parts of the dental flask we put Vaseline as separating medium to make it easily to separate dental flask from dental plaster. Then we mixed dental plaster according to manufacture instructions. After waiting for 3 min we put the metal discs that contain wax discs on the sandblasted surface in the dental flask, every dental flask contains 10 metal discs which means one subgroup in every flask.

Figures (14), (15) dental plaster poured into the dental flasks**.**

We wait plaster to dry for15 min then we put two layers of separating medium (IsoLant/C.M.S. ENGLAND) first we add the first layer and wait to dry then we added the second layer. After that we put the upper part of dental flask and complete plaster pouring and close the flask under pressure to allow to the excess material to come out by using flask press device. When the plaster completely hardened, the dental flask was put in the melting machine (META MD-145 AUTOMATIC WAX MELTING DEVICE) in boiling water for 15 min to insure that wax discs completely melted. Then we open the flasks and the two parts were washed with hot water to remove all the residual wax, allowed to cool dawn at room temperature for about 10min.

Figure (16) META machine for wax melting.

Figure (17) the shape of the specimens after wax melted.

Figure (18) Applying of separating medium**.**

Then we applied separating medium (IsoLanT/C.M.S. ENGLAND) in the upper part of dental flask especially in the molds and for lower part just around the metal discs [did not put it on the metal discs] for the discs we applied uniform coating of metal primers by using small brush (ALLOY PRIMER – KURARAY; Z PRIME PLUS – BISCO) and leave it to dry in the air for 5 min according to manufacture instructions. The active part of alloy primer is Thionephosphate (VBATDT) and phosphoric acid monomer (MDP).And for Z Prime plus the active part is a combination of two active monomers, MDP, a phosphate monomer, and BPDM, carboxylate monomer.

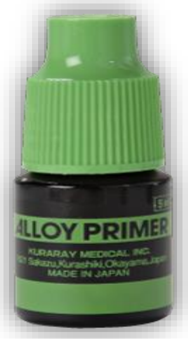


Figure (19), (20) Alloy primer –kuraray.

Material	Composition	Indication	Operating Procedures
Alloy Primer(AP)	MDP 10-Methacryloyloxydecyl dihydrogen phosphate and VBATDT 6-(4-Vinylbenzyl-N-propyl) amino-1,3,5-triazine-2,4-dithione, in acetone	1-Adhesion of metal inlays, Onlay, crowns and bridges Adhesion of metal cast posts. 2-Repair of fractured resin-based facing crowns and porcelain-fused metal crowns and bridges. 3-Fabrication of removable dentures with metal base. Clasp or attachment. 4-Repair of dentures.	1-treating the metal frame. 2-sandblast the metal frame. Then, wash the surface with water. 3-Applaying ALLOY PRIMER to the metal adherent surface with the sponge and leave it to drying. 4-Curing the denture base acrylic resin.

Table (3) Alloy primer used in this study.

Figure (21), (22) Z-prime plus –Bisco.

Material	Composition	Indications	Operating Procedures
Z-Prime Plus	MDP(Phosphate monomer), PPDM(Carboxylate monomer)	Used as surface treatment for the following restoration 1-Zirconia. 2-Alumina. 3-Metal\Alloy. 4-composite. 5-Endodontic Post. 6-Intraoral repairs.	1-Clean the adherent surface of metal, using one or more of the following options: pumice scrub, ultrasonic cleaning, steam cleaning and\or sand blasting using aluminum oxide(30-100) micron grit at 30-45psi. 2-Applay 1-2 coats of Z-PRIME PLUS, uniformly wetting the bondable surface. Dry with an air syringe for 3-5 seconds. 3-curing the acrylic resin.

Table (4) Z-Prim Plus used in this study.

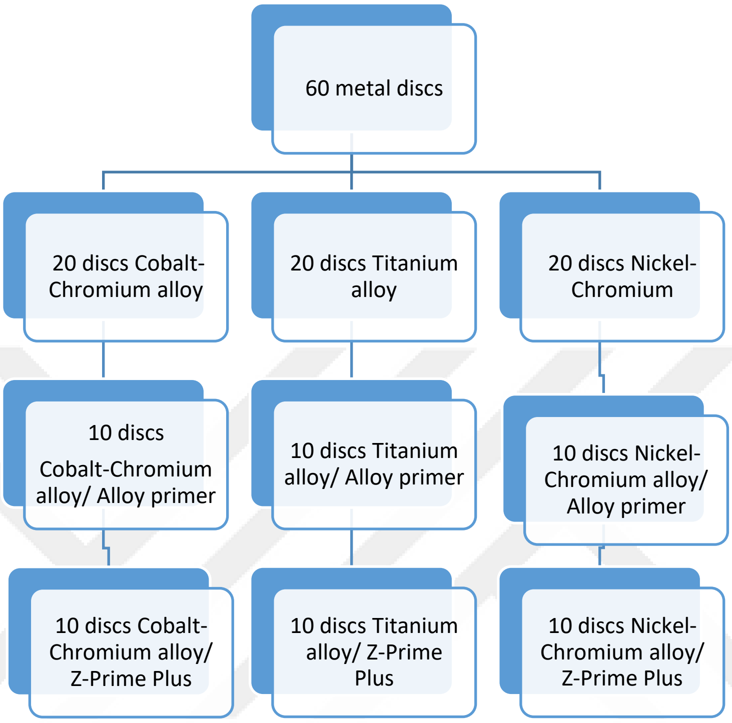


Figure (23) low-chart illustrate all the groups in the test

Heat cure acrylic resin material (Meliodent, GERMANY) was mixed and packed in to the plaster molds first by trial packed at 1000 psi, then opened to remove the excess material. A second pack has done at 2000 psi and held in the place for 5 min. The heat cure acrylic resin was processed for 60 min as manufacturing instructions, the polymerization started at room temperature in the water bath of the machine over the first 30 min the water was heated up to 100° C, then the flask held at 100° C for the rest time 30 min to complete the polymerization, and then took it out from the machine and left it to cool at room temperature.

Figure (24) Meliodent heat cure acrylic resin.

Figure (25) packing of heat cure acrylic resin.

Figure (26) dental flask.

Table (5) Heat cure acrylic resin used in this study.

Material	Composition	Indications	Operating Procedures
Meliodent heat cure acrylic resin.	Powder: Methyl methacrylate-Copolymer. Liquid: Methyl methacrylate-Dimethacrylate.	Dental resin for making dentures. For Fixed and removable prosthetic devices.	Use hard type of stone for making the model and embedding it. Meliodent Heat Cure can be mixed individually however, the recommended mixing ratio is 35g powder: 14ml liquid. Pour the amount of liquid required into a mixing bowl, and add powder carefully, until it does not soak up any more liquid, and the surface of the powder remains dry. Remove the excess powder and knead the dough thoroughly with a spatula for 1

			minute. Meliodent Heat Cure achieves a consistency suitable for packing after approx. 10 mins. The processing time is approx. 25mins, but can vary depending on the room temperature.

All the sixth groups are ready to put in the self-cure acrylic resin. Group were as following: group 1: Cobalt-Chromium \ Alloy primer, group 2: Cobalt-Chromium \ Z-Primer plus, group 3: Titanium \ Alloy primer, group 4: Titanium \ Z-Primer plus, group 5: Nickel-Chromium \ Alloy primer, group 6: Nickel-Chromium \ Z-Primer plus. After we complete the processing of heat cure acrylic resin, we used low speed handpiece to remove all the excess of material. All the finished specimens were immersed in water at 37°C for 24h.

Figure (27) specimens after processing of acrylic resin.

Figure (28), (29) low speed handpiece and burs.

Figure (30) the final shape of samples after finishing.

Autopolymerizing acrylic resin (Imicryl, TURKEY) was used to prepare metal disks to put into the universal testing machine. All the samples were embedded in self cure acrylic resin by using metal (12mm in light, 20mm in diameter).

Figure (31) self-cure acrylic resin**.**

Table (6) Self-cure acrylic resin used in this study.

Material	Composition	Indications	Operating procedures
Self-Cure Acrylic resin, IMICRYL	Powder: Polymethylmethacrylate. Liquid: Methyl methacrylate	Using reforming and prosthesis repairs.	The acrylic dough is prepared in an adequate container. The polymer is poured over the monomer in the indicated ratios.

	The mixing is continually made crosswise during 30 seconds approximately in order to ensure the complete incorporation of polymer and monomer particles. Put a lid on the Co Cover the container to avoid the inclusion of air until the mixture reaches a thickness in a fluid phase. Finally, Immediately empty the mixture in the corresponding area.

Figure (32) metal mold

3.3 Thermocycling

All the samples were thermal cycled in Salibrus-Technica, Turkey for 5000 times which nearly stimulated 4 years of clinical use. The temperature was between 5° C to 55° C with a dwell time 30seconds for each temperature and transferring time from one bath to the other was 10

Figure (33) Salibrus-Technica for thermocycling

Then all the samples were fixed in self-cure acrylic resin by using a special stainless steel mold, after that we put the specimens in the universal testing machines to measure the shear bond strength (SBS).

Figure (34) the samples inside the self –cure acrylic resin**.**

Figure (35), (36) all the 60 samples are ready to measure SBS

Shear bond strength was measured by using the universal testing machine (Instron 3345 model, USA)We used a crosshead speed of 0.5 mm/min, the knife edge blade of the machine was parallel to the interface of heat cure acrylic resin and metal surface, the de bonding of shear bond strength was registered in Newton (N). And the failure load (N) was divided by the bonding area (mm^2) then the debonding shear strength was converted into MPa.

Stress (MPa) = failure load (N)\Surface area (mm^2).

Figure (37) universal testing machine**.**

4. 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), one-way ANOVA was used in the comparison of groups, post Hoc Tukey multiple comparison test was utilized in the comparison of two groups. Statistical significance level was established at $P<0.05$.

5. RESUTS

Table (7) shear bond strength means for all metal alloys groups (MPa).

	Number(n)	Shear Bond Strength (MPa)	Shear Bond Strength (N)
Cobalt-Chromium	20	1,85±0,26	71,25±9,86
Titanium	20	13,18±4,59	507,42±176,61
Nickel-Chromium	20	6,02±5,16	231,71±198,69

Tukey HSD Multiple Comparison Test	Shear Bond Strength (MPa)	Shear Bond Strength (N)
Cobalt-Chromium**/** Titanium Alloy	0.0001	0.0001
Cobalt-Chromium/ Nickel-Chromium	0.0001	0.0001
Titanium / Nickel-Chromium	0.0001	0.0001

There was a statistically significant difference between the Shear Bond Strength of Cobalt, Titanium and Nickel groups (p=0.0001). The Shear Bond Strength of the Titanium group was statistically significantly higher than the Shear Bond Strength of the Cobalt and Nickel groups (p=0.0001). The Shear Bond Strength of the Nickel group was found to be statistically significantly higher than the Shear Bond Strength of the Cobalt group (p=0.0001).

Table (8) Shear Bond Strength means for Primers groups (MPa)

	Number (n)	Shear Bond Strength (MPa)	Shear Bond Strength (N)
Alloy Primer	30	4.22±3.87	162.6±148.89
Z Prime Plus	30	9.81±6.75	377.65±259.62

The Shear Bond Strength of the Z Prime Plus group was statistically significantly higher than the Shear Bond Strength of Alloy Primer group (p=0.0001).

Table (9) Two-way analysis of variance for Shear Bond Strength.

Source	Type Ⅲ Sum of Squares	df	Mean Square	F	P
Metal Alloys	1314.44	2	657.22	220.09	0.0001
Primer	468.39	1	468.39	156.85	0.0001
Metal Alloy* primer	278.16	2	139.08	46.58	0.0001

There was a statistically significant difference between the Shear Bond Strength of Metal Alloys groups (p=0.0001).
There was a statistically significant difference between the Shear Bond Strength of Alloy Primer groups (p=0.0001).
There was a statistically significant difference between the Shear Bond Strength of Metals*Alloy Primer groups (p=0.0001).

Table (10) Shear Bond Strength of Alloy Primers

	Number (n)	Shear Bond Strength (MPa)	Shear Bond Strength (N)
Co-Cr/Alloy Primer Group (1)	10	2.04±0.22	78.43±8.45
Co-Cr / Z Prime Plus Group (2)	10	1.66±0.11	64.06±4.37
P		0.0001	0.0001
Ti - Alloy Primer Group (3)	10	9.44±1.55	363.29±59.81
Ti - Z Prime Plus Group (4)	10	16.93±3.3	651.55±126.89
P		0.0001	0.0001
Ni-Cr- Alloy Primer Group (5)	10	1.20±0.22	46.08±8.64
Ni- Cr - Z Prime Plus Group (6)	10	10.84±2.13	417.34±81.82
P		0.0001	0.0001

The Shear Bond Strength (MPa) of group 2 was statistically lower than The Shear Bond Strength of group 1(P=0.0001).

The Shear Bond Strength (MPa) of 4 group was statistically higher than The Shear Bond Strength of3 group (P=0.0001).

The Shear Bond Strength (MPa) of group 6 was statistically higher than The Shear Bond Strength of group 5(P=0.0001).

Table (11) Shear Bond Strength Alloy Primer

	Number (n)	Shear Bond Strength (MPa)	Shear Bond Strength (N)
Group 1	10	2.04±0.22	78.43±8.45
Group 3	10	9.44±1.55	363.29±59.81
Group 5	10	1.20±0.22	46.08±8.64
P		0.0001	0.0001

Turkey multiple comparison test	Shear Bond Strength (MPa)	Shear Bond Strength (N)
Group 1and Group 3	0.0001	0.0001
Group 1 and Group 5	0.119	0.119
Group 3 and Group 5	0.0001	0.0001

There was a statistically significant difference between the Shear Bond Strength (MPa) of Group 2, Group 4 and Group 6 (p=0.0001).

The Shear Bond Strength (MPa) of Group 2 was statistically significantly lower than the Shear Bond Strength (MPa) of Group 4 and Group 6 (p=0.0001). The Shear Bond Strength (MPa) of Group 4 and Group 6 was statistically significantly higher than the Shear Bond Strength (MPa) of the Shear Bond Strength (MPa) of (p=0.0001).

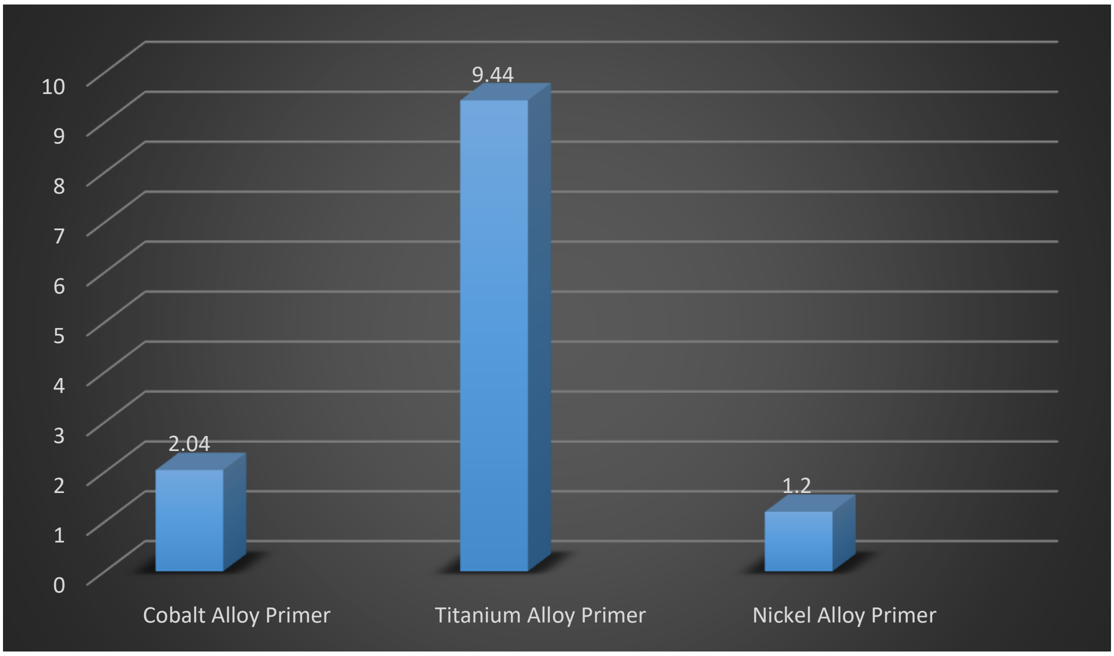


Figure (38) Bar-Chart for the Shear Bond Strength for Alloy Primer.
Cobalt =2.04, Titanium= 9.44, Nickel = 1.20 (MPa)

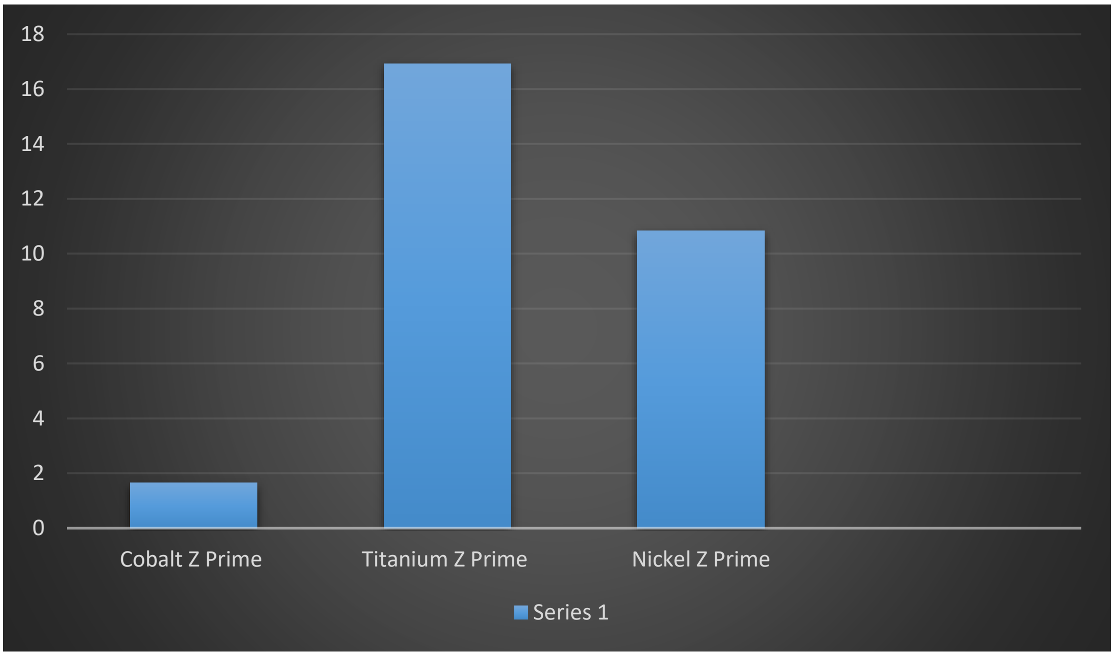


Figure (39) Bar-Chart for the Shear Bond Strength for Z Prime Plus.
Cobalt= 1.66, Titanium= 16.93, Nickel = 10.84 (MPa)

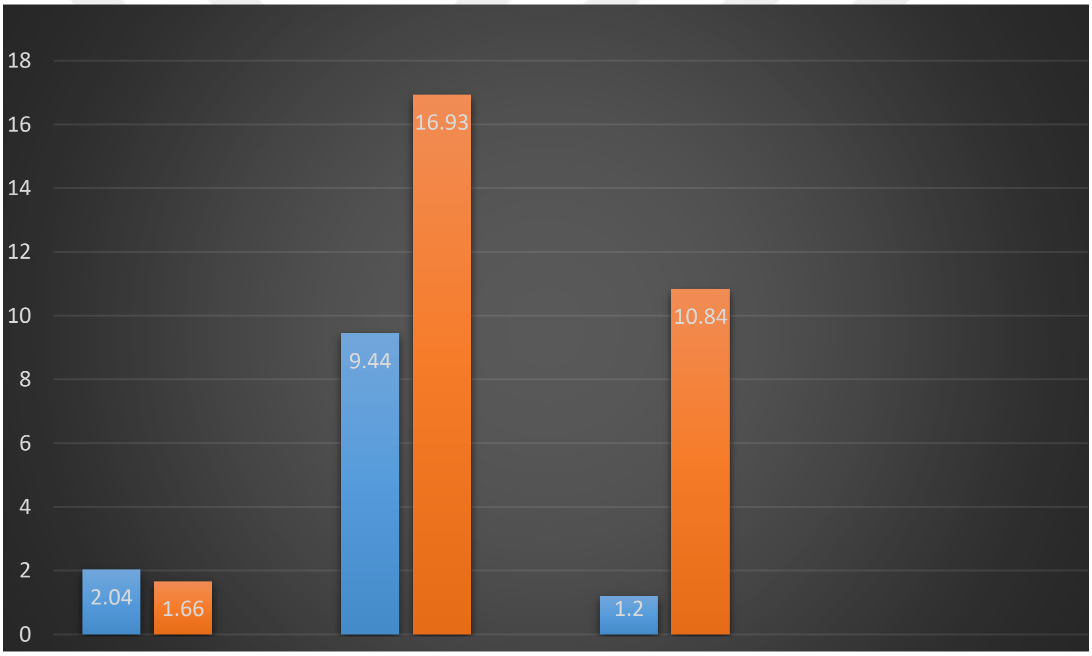


Figure (40) the effect of Alloy Primer and Z Prime Plus on the Shear Bond Strength of Cobalt, Nickel and Titanium alloys.

6. DISCUSSION

Removable partial denture is considered as a good treatment option for patient who cannot afford the other types of dental treatment as a crown or fixed bridge or implants not only because it is more economical than bridge or dental implant. RPD can replace missing oral structures as well as support the remaining teeth. That means the purposes of using partial denture are to maintain the remaining oral structure, improve phonetics, establish dental relationship, increase masticatory efficiency and enhance the required esthetics. [13]The common base metal materials that are used in RPD fabrication are Cobalt-Chromium alloy, Nickel-Chromium alloy and Titanium alloy. In lieu[19] suggests the trend for removable prosthesis is using cobalt-chromium frameworks or implant supported fixed prosthesis, this part of dental materials are important for prosthodontic research.

The bonding between the heat cure acrylic resin and the metal alloys as Cobalt-Chromium, Titanium alloy and Nickel-Chromium have been improved by using different methods which are called surface treatments. These procedures are classified as roughening of the surface to produce micromechanical retention or chemical bonding or both of them, or by using a chemical etching, [42] sandblasting, electrolytic etching. [43]

A failure of the bond at acrylic resin denture base and metal alloy framework can result in prosthetic failure by creating space between the metal and PMMA. Whereas stain, microorganism and oral debris can accumulate which increase the risk for infection and fracture of prostheses. [44]

Titanium is a new material introduced into RPD construction because it has excellent biocompatibility, sufficient corrosion resistant, good strength, does not cause any allergy, easy to use it and nontoxic. The desirable physical and mechanical properties of the Ti-6Al-4V a commercially pure titanium (CP) are very similar to each other. [44]Yamauchi et al. examined the results of patients who had used dental prosthesis constructed from commercially pure titanium. [45] They found that, there were no clinical problems with commercially pure titanium when used as partial denture framework. However, there was some discoloration at the finish line due to stain penetration, despite of using adhesive resin that contains 4-META (META-DENT, Sun Medical, Kyoto, Japan) as a denture base. [44]

During the study of this phenomenon, Matsumura et al. found that the shear bond strength of adhesive resin that contains 4-META to pure titanium decreased after thermocycling. [46] Usually there is a titanium oxide layer cover the surface of pure titanium metal which is stable in an atmospheric environment. That means after adding of metal primer, the metal primer will adhere to this layer rather than adhere to CP titanium itself, and the chemical bond can result from reaction of metal oxide with phosphoric acid or carboxylic acid derivatives.[44]The CP titanium framework is more flexible than the Cobalt-Chromium alloy and during function the debonding can occur between titanium frameworks and denture base. Therefore titanium framework must be fabricated to be hard enough to make the denture deflection as minimum as possible. [44] It has been reported that the sandblasting procedure makes the alloy- water contact angle more smaller and greater wettability. [51]And creates a roughened surface to increase the mechanical interlocking for acrylic resin by removing the contaminated layers, [53] and provide a great surface area for the bond. Metal sandblasting with Al_2o_3 particles produce a passive film from Ni, Co, Cr oxides. [52]According to Ohno et al. [54] who defined the passive film of hydrated chromium oxy hydroxide and made a general formula for this layer: Crx(OH) $_{3.2}$ nH_2O. This film has the ability to be formed on the surface metal alloy that contains Ni, Co, and Cr and on the stainless steel. The air born–particle abrasion (APA) is one of the surface treatment that used to increase the increase the bonding strength of metal alloys. [54]

Giachetti et al. [58] used the scanning electron microscopy (SEM) to make a morphological analysis of sandblasted metal surface with different sizes of alumina particles (50µ vs150µm). The result showed that the surface treated with 150µm alumina particles presented deep and large cavities where the resin can penetrate more compared to the surface treated with 50µm which appeared irregular and rough.

Kern and Thompson [59] evaluated the surface morphology after titanium alloy treated with sandblasting with 110µm alumina particles. 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. However, the result of study that done by Groro N [60] shown that the ultrasonic cleaning of sandblasted surface will decrease the adhesive strength of luting resin. Therefore the ultrasonic cleaning after specimen surface treated with the sandblasting should be avoided. After sandblasting a new surface layer will be formed this layer is high in activity and purity.[61] A highly active a new surface has a high ability to chemical bond, which also means it is suitable to contamination during the ultrasonic cleaning process , then the bonding surface will lose its bonding ability to the resin materials and the bonding strength will decrease.

To get a good result of this technique there are some parameters in the air borne particle abrasion with alumina, the first parameter the propulsion pressure from 0.05 to 0.45 MPa, the distance from the specimen surface and the nozzle range from 5 to 20 mm and the last parameter is the time for exposure from 5 to 30 seconds. [55, 56]

In 2001, Yoshida, et al [47] clearly expressed that the using of the Alloy primers that contains VBATDT and MDP as an active group will increase the bond strength between the base metal alloys and the acrylic resin. Base on the fact that, MDP has an ester phosphate group which provides a good chemical bond between the metal oxide (Cr_2O_3) layers that covered the surface of the Cobalt-Chrome, which gives a good union of the cements to base metal alloys. [48]Mercapto groups in the VBATDT monomer react with the noble metal alloys and produce a chemical bond between the metal-resin interfaces.

Accordıng to Antoniadou etal [49] (2000) Using of the alloy primer is fast, simple and a good way to increase the durability of the shear bond strength between the acrylic resin and metal alloys. However this bond depends on the composition of the metal alloy.Bulbul and Kesim[3] studied the effect of three types of metal primers on the shear bond strength of three acrylic resins to different types of alloys. The primers were Meta Fast which contains 4-methacryloxyethyl trimellitate anhydride (4-META), Metal Primer which contains Phosphoric acid MDP and Alloy Primer which contains 6-(4-vinylbenzyl-n-propyl) amino-1, 3, 5-triazine-2, 4-dithione (VBATDT) and phosphoric acid MDP. Four groups were evaluated for resin and alloy. One group as control group and the other three groups bonded with the three different primers. After thermocycling, the result of the shear bond strength was vary according to the type of primer and metal that used. This study suggested that primers should be selected according to the type of metal alloy that will be used.

The authors recommended that, the use of Meta Fast for titanium alloys, Metal Primer with base metal and Alloy Primer for noble alloys. They explained that the primer that include Mercapto groups in the VBATDT monomer react chemically with noble alloys and increase the bond at the metal-resin interface. Additionally the primer containing MDP monomer provide a good strength with the Co-Cr alloys better than the primer that contains 4-META monomer.

Mohsin A [63] compared the shear bond strength of sandblasted Ingot and cast Co-Cr alloy to heat cure and self-cured repair acrylic resin after treating with different chemical composition of metal primers. The author concluded that, the using of any of alloy primers will increase the durability of clinically bond strength of RPD framework more than non-primed framework. And Z-Prime Plus treated groups had the highest bond strength regardless of type of resin and alloys. The high bond strength could be due to the presence of phosphate monomer in the Z-Prime Plus as the active part, which is chemically bond to the methacrylate group at one side and phosphoric acid group on the other side.

Storage conditions and its time, for bond strength tests the distilled water, 0.5% chloramine-T, saline, 2% glutaraldehyde, 0.05% saturated solution of thymol and 10% formalin solutions were studied as storage media,[64, 65]storage in sodium hypochlorite (NaClO) resulted in lower bond strength.[59]In our study we choose the distilled water as storage media before the test. To stimulate clinical condition Thermal cycling has been used. Mair found that oral cavity temperature changes from-4℃ to 0℃ when eating ice cream and between 60℃ - 65℃ when eating a hot cheese sandwich. [67] As most of the time dental restorations are subjected to a little temperature variation, thermocycling may not have a significant effect but it can lead to spontaneous debonding of specimens. [68]

A short regimen of thermal cycling is recommended by ISO TR 11450 standard (2003) is 500 cycle. [69]According to the previous studies the number of cycles was decided 6000 thermal cycles are equal to 5 years of clinical use.[70, 71] so 5000 cycles that is used in our study are equal to 4 years of clinical function. Celik et al. [72] 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, from all the aging methods. The bond strength was remarkably reduced after thermocycling. 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. [72] Most of in vitro studies used monotonic tests as compression, shear or tensile strength to examine the mechanical properties of the dental materials. [73]Because of the previous tests cannot produce fatigue damage as that happened in the mouth, the studies with fatigue tests should be done to get better clinical results. [74]Oilo[75]classified the bond strength test into quantitative tests and qualitative tests. The quantitative tests study predict the lifetime of the bond and the load capacity and qualitative tests study bond failures. [75]

The bond strength can be measured by clinical performance and laboratory methods. [76] The nominal bond strength is calculated as the following maximum applied force / the bonded cross-sectional area. [77] It is possible that there will never be one laboratory test that will predict accurately the clinical performance of the dental materials. [78] These laboratory tests can be dynamic or static test. [79]

In the static tests, force applied when the specimen is in stationery state not like dynamic tests where the sample is in dynamic state. The static tests are classified into micro-tests where the bond area is <3mm^2 bond area and macro tests with >3mm^2. [76] The macro bond strength can be measured in tensile, shear or using a push-out protocol. [80]In our study the bonded area was >3mm so the test was macro bond strength test.

In the shear bond test (SBS), there are two materials connected to each other by adhesive agent and loaded in shear until fracture happens. It is a widely used test. [81] Some authors recommended a mandatory shear bond strength about 20 MPa for permanent success of the repaired restoration. ISO 10477 for polymer-based materials is recommended shear bond 5 MPa. [82]Regarding to the results of our test all the tested groups fulfilled the ISO requirement of 5 MPa except group1 (Cobalt-Chromium\Alloy primer), group2 (Cobalt-Chromium\Z prime plus) and group 5(Nickel-Chromium\Alloy primer). The shear bond strength of the acrylic resin and metal alloys after using of the metal primers was tested in most of the previous publications ranged from 2.48 to 18.70MPa.[45, 52] Concerning the results of the present study the shear bond strength of all tested groups was in these range except of Cobalt-Chromium / Alloy primer (Group1), Cobalt-Chromium / Z-Prime Plus (Group 2) which were 2.04±0.22, 1.66±0.11MPa and Nickel-Chromium / Alloy primer (Group 5) was 1.20±0.22 MPa. The other results of the current study are according to the ISO requirement of 5 MPa as Titanium \ Z-Prime Plus (Group 4) 16.93±3.3 MPa, which was the highest bond strength in our study, then Nickel-Chromium \ Z-Prime Plus (Group 6) with 10.84±2.13 MPa. Titanium \Alloy Primer (Group 3) 9.44±1.55 MPa.

Bulbul et al, evaluated the effect of metal primers on the shear bond strength of acrylic resins to three different types of metals. The highest mean SBS values achieved by the base metal alloys specimens (2.48±0.7MPa), may depend on the surface roughness and thickness of the metal oxide layer of the alloy surface. [44]Also the bond strength to the Cobalt-Chromium was improved after using of metal primers that include MDP monomer due to formation of Chromium Oxide (Cr_2O_3) on the surface. This primer demonstrate better bond strength than do the primers contain 4-META monomer.[52]We evaluated the effect of alloy primer on the shear bond strength between the base metal alloy and heat cure acrylic resin after thermocycling for 5000 cycles. In the result there is significant effect of the alloy primer on SBS. The SBS of Alloy Primer increased 4.22±3.87 MPa and for the Z-Prime Plus was 9.81±6.75 MPa. For the metal alloys was Titanium 13.18±4.59, Nickel-Chromium 6.02±5.16, and Cobalt-Chromium 1.85±0.26 MPa. Which means there was a significant effect of using alloy primers to increase the bond strength of heat cure acrylic resin to base metal alloys. The highest value was for Titanium alloy after treated with Z-Prime Plus 16.93MPa than Cobalt-Chromium \ Alloy Primer 2.04MPa. Although, in the previous study the Cobalt-Chromium\ Alloy Primer specimens had the highest value of the SBS may be due to the composition of the Alloy Primer. Which contains MDP monomer and the type of the chemical bond that formed between this monomer and chromium oxide (Cr_2O_3) and thickness of this layer. Also the Z-Prime Plus contains phosphate monomer as well as carboxylate monomer. Which reacts chemically with the methacrylate group on one side and with the phosphoric acid group on the other side. These results show similarities with the study result of Ali M el at.[63]

Sandeep et al, investigated the effect of sandblasting and metal primer on the SBS between Co-Cr alloy and heat cure acrylic resin. [42]The result was that the SBS improved after metal surface treated with sandblasting and metal primers. The result obtained in our study also indicated similar findings. The bond strength values obtained in the previous study were comparable to the bond strength values obtained in the similar studies reviewed in the literature. Kim et al [53] the bond strength values of primed and sandblasted groups were 17.1 MPa and 18.70 MPa. Which were higher than our result means SBS for Z-Prime Plus 9.81±6.75 although our samples were treated with sandblasting then primed.

6.1. Discussion of results

Group 1: Co-Cr / Alloy primer

In our study the SBS result of (group 1) Co-Cr / alloy primer (2.04±0.22) was higher than (group 2) Co – Cr \ Z-Prime Plus (1.66±o.11). Which is nearly similar to the result that has been done by Mehmet B, [3] when he compared the effect of the alloy primer on SBS between heat cure acrylic resin and Co-Cr alloy. This could be due to using the same type of heat cure acrylic resin (Meliodent Rapid Repair) and doing same number of thermocycle (5000 cycle and temperature between 5℃& 55℃). The small difference in the score (1.30± 0.29 MPa) might be due to the difference of the Co-Cr alloy composition .Also high SBS result of Co-Cr alloy was obtained by Kalra S el al,[1] for the samples that treated with Alloy primer (18.70MPa) it was higher than our score because in this study they did not do thermocycling which decreases the value of SBS by aging.

Group 2: Co- Cr /Z-Prime Plus

In the current study the SBS of Co-Cr alloy after treated with Z-Prime Plus was 1.66±0.11MPa. However this result was dissimilar to the results showed by Mohsin Ali [63] which was 12.990 (Mean). This high result may be due to doing the test without thermocycling. Also they apply Z-Prime Plus after treating the specimens' surface with 600-grit silicon carbide discs under water, instead of sandblasting. The high SBS scores could be due to phosphate monomer MDP which bond chemically to methacrylate group at one side and the other with phosphoric acid group. In our study we use both of them sandblasting to increase surface roughness, and thermocycling which decrease the value of SBS.

Group 3: Ti / Alloy primer

In our study, SBS of treating Ti alloy with Alloy primer was 9.44±1.55 MPa. Which is supported by Taira et al (1998) study when MDP containing primers (alloy primer) and Ti alloy are used higher bonding strength was obtained. Because these primers have a fine oxide layer, which is responsible for this high bond. On the other hand, SBS of CP Ti according to Kawaguchi T et al,[84]after they treated with Alloy primer then subjected to thermocycling for 10,000 cycle the result was higher than our result. It was 14.4 MPa. Although they did longer thermocycling than us the difference in the type of Ti alloy composition may be the reason for the difference of the result.

The CP Ti contains >99.0 % Ti that means the bonding between oxide layer of CP Ti and phosphoric acid of MDP will be stronger than the bonding of this acid with Ti90Al6v4 alloy. Also our results were similar when compared with Veljiee T M et al [86]'sscore which was 7.593±o.245. These two studies have similar protocols (sandblasting protocol, thermocycling number and temperature) but the only difference was they used CP Ti but we used Titanium alloy.

Group 4: Ti / Z-Prime Plus.

This group showed the highest SBS (16.93±3.3) among other tested groups. This could be the presence of MDP as a component in Z-Prime Plus. Which supports what Taira et al [87] (1998) reported, the presence of a fine oxide layer on the Ti alloy surface with MDP containing primers will produce high SBS.

Group 5: Ni-Cr / Alloy Primer.

This group demonstrates low value of SBS (1.20±0.22 MPa). There is no big difference between this score and Co-Cr / Alloy Primer score's (2.04±0.22) we think this low score in the SBS could be due to weak chemical bond formed between heat cure acrylic resin and metal alloy. Salonga et al [(85)] studied effect of concentration of Chromium content in Nickel-Chromium alloys when he connected two metal discs by using luting agent which is methyl methacrylate-based resin (MMA). The SBS result after 20,000 cycles was high for pure chromium (50.6 MPa) which is higher than SBS of pure Nickel (24.7 MPa). This high scores may be due to the chemical bond was between two metals not between metal and acrylic resin.

Group 6: Ni-Cr / Z-Prime Plus.

The SBS of this group was 10.84±2.13. Which is considered a second high SBS score between the tested groups. Although there is a small differences in composition of Cobalt-Chromium and Nickel-Chromium, the SBS results of group 6: Ni-Cr / Z-Prime Plus was higher than group 2**:** Co- Cr /Z-Prime Plus (1.66±0.11 MPa).There was no previous study using Nickel-Chromium alloy with Z-Prime Plus primer is similar to our group number 6. But Mohsin Ali et al studied Z-Prime Plus primer with Co-Cr. If we compare our results with this study because of the similar composition of Ni-Cr and Co-Cr we can say that their results are similar with ours. [(63)] Co-Cr group results treated with Z-Prime Plus was12.990 MPa. The small difference may be because they did not do thermocycling which decreases SBS scores.

6.2. Comparison of primer types

According to our result Z-Prime Plus had higher SBS scores 9.81±6.75 MPa than Alloy primer 4.22±3.87 MPa, especially when used with Titanium alloy and Nickel-Chromium alloy.

This could be due to the chemical bonding between oxide layer of these metal alloys and active parts of Z-Prime plus (MDP and PPDM) which is not present in the Alloy Primer. Limited information is available about the bond durability between heat cure acrylic resin and Ni-Cr alloys and Z-Prime Plus. Alloy primer had low scores for Co-Cr and Ni-Cr alloy.

The dimension of the test specimens may not represent the actual clinical condition and the difference in the geometry may affect the distribution of the stress which may affect the SBS scores. In our study the storage medial was the distilled water. However, there is saliva in the oral cavity instead of distilled water, and they are not chemically similar. Also the effect of dynamic forces cannot be tested with a chewing stimulator. The bond strength of heat cure acrylic resin to metal alloy is sensitive to mechanical and chemical influences in the oral cavity. Even we did thermocycling we cannot simulate every condition occurring in the mouth. The study can be improved by using much more number of samples, metal alloys different alloy primers and different types of resin.

7. CONCLUSION

Within the limitation of our in vitro study, we can conclude that:

1. Titanium alloy showed the highest SBS scores among the metal alloys tested (13.18±4.59), whereas the Cobalt-Chromium alloys showed the lowest scores (1.85±0.26).
2. Z- Prime Plus demonstrated higher SBS scores (9.81±6.75) than Alloy primer (4.22±3.87).
3. The metal primer should be selected depending on the type of the metal alloy. For Titanium and Nickel-Chromium using Z-Prime Plus as a metal primer (16.93±3.3 MPa, 10.84±2.13 MPa) showed better result for resin to metal bonding.

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DEDICATION

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