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Research Article

The Effect of Different Metal Primers on Shear Bond Strength Between Heat Cure Acrylic Resin and Different Metal Alloys after Thermocycling

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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.

Materials and Methods: Total number of 60 disc-shaped wax patterns (10 mm in diameter and 3 mm in thickness) were cast in cobalt-chromium, nickel- chromium, and titanium alloys (Ti90AI6V4). After casting, the discs surface was sandblasted with 110 µm aluminum oxide for 5 seconds. Specimens of each metal were divided into two groups (n = 10) and received either Alloy Primer (Kuraray, Japan) or Z_Prime Plus (Bisco, Schaumburg). The groups thus consisted of Group 1: 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 distilled water at 37°c for 24 hours after polymerision prior to thermocycling (5000 cycles at 5 c°- 55 c° with a 30 seconds dwell time for each temperature). After thermocycling, the specimens were tested in an universal testing machine at a cross speed of 0.5mm/min in shear mode. Data (MPa) were analysed using one-way ANOVA and post Hoc Turkey (P ⍰ 0.05).

Result: The one-way ANOVA indicated that SBS values varied according to the type of metal and metal primers used (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) For metal primer the shear bond strength of the Z-Prime Plus groups (9.81 ± 6.75 MPa) was statistically significantly higher than 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 bond of heat cure acrylic denture base resin to Titanium alloy.

Keywords: *Shear Bond Strength; Titanium; Cobalt Chromium; Nickel Chromium Alloys; Thermocycling; Metal Primer*

Abbreviation

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; **μ**m: 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); and Others; MMA: Methyl Methacrylate.

Introduction

Cobalt-chromium alloy (Co-Cr) and commercially pure titanium (CP Ti) are commonly used to fabricate the metal framework of removable partial dentures, due to their good mechanical properties. Heat cured acrylic resin is used as denture base polymer for removable prostheses [1,2].

For durability of the prosthesis the bond between the denture base resin and the metal components must be adequate to prevent microleakage and bond failure due to different coefficients of thermal expansion of the heat cure acrylic resin and the metal framework [2] and polymerisation shrinkage of heat cure acrylic resin [1,3,4].

The introduction of chemical metal-resin bonding systems, such as 4-methacryloy-ethyl trimellitate anhydride (4-META), significantly improved the bond between resin and metal [1,2,5-9].

Traditionally mechanical retention such as mesh, lattice or bars held the denture base resin on the framework. Contemporary techniques include chemical etchants, tinplating and silica coating [4,6,9-11]. The primer system was easier and more economical because it is not a sensitive technique and it doesn't need special equipment [4]. Chemical bonding of the resin to metal is more desirable than mechanical retention in RPD construction. Several adhesive primers, including Metal Primer (Ivoclar Vivadent AG, Schaan, Liechtentein), containing 10-methacryloxydecyl dihydrogen are available for RPDs and resin bonded prostheses [1,12].

This study was designed to evaluate the effect of two types of metal primers along with sandblasting on the bond strength and bond durability between heat cured acrylic resin and three different metal alloys after thermocycling. It was hypothesized that the bonding strength durability between heat cured acrylic denture base and base metal would be improved by using the priming agents.

Materials and Methods

Preparation of the specimens

The materials that used in this study are listed in table 1.

Brand	Manufacturer	ISO Number
Cobalt-Chromium alloy	ELEKSAN Metaplasm 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 Gruner Weg 11 63450 Hanau, Germany	ISO 10993 and ISO 7405
Self -Cure Acrylic Resin	IMICRYL, TURKEY	ISO 13485:2003

Table 1: *Materials used in the test.*

Disc-shaped metal alloy (3 mm in thickness and 10 mm in diameter) were cast using Cobalt-Chromium alloy (Metaplus UNI, Eleskan, Germany), Nickel- Chromium alloy (Solera VK, Germany), Titanium alloy (Dent Index, Turkey), according to the manufacturer's instruction.

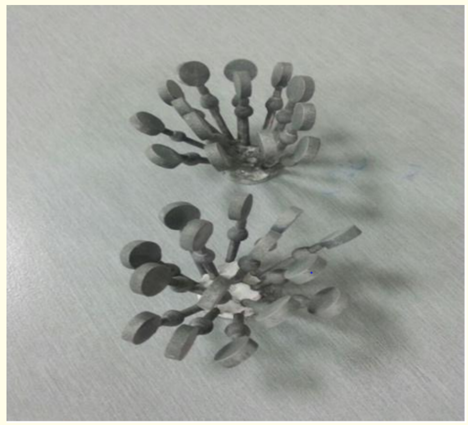
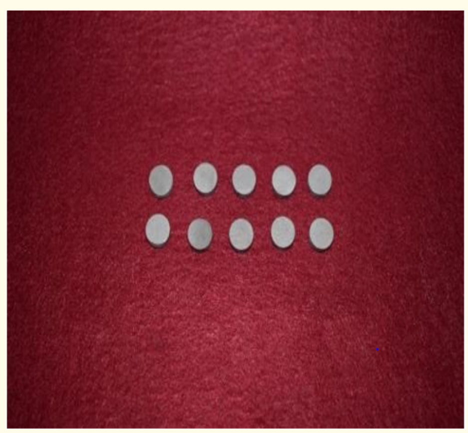


Figure 1: *Metal disks after casting.*



Figures 2: *Metal discs after finishing.*

The total number of samples was 60 metal discs divided into three groups (n = 20) according to the type of metal alloys. Then divided into subgroups (n = 10) depends on metal primer that used. After casting, the disc-surfaces were subjected to Aluminum oxide with 110 mm particles size for 5 seconds at 80 psi, at 45-degree angle. There was 5 mm distance from the metal surface and the nozzle of the machine (Star Dental, TURKEY).

Figure 3: *The final shape of metal discs after sandblasting.*

For heat-polymerized resin 60 wax discs (7 mm in diameter and 2.5 mm in thickness) were prepared using silicon ring. The wax discs (baseplate wax, Cere Wax, TURKEY) were placed in the center of disc-shaped metal specimens by dropping technic. Specimens were invested in brass flasks (10 samples per flask). After isolation of the lower part of the flask with a separating agent (IsoLant/C.M.S. ENGLAND) When the plaster hardened, the flask was placed in boiling water for 5 minutes. Flask opened, and the two parts were washed with hot water to remove the residual wax. The upper member was isolated with a separating agent (IsoLant/C.M.S. ENGLAND), and the flask was cooled in room temperature air.

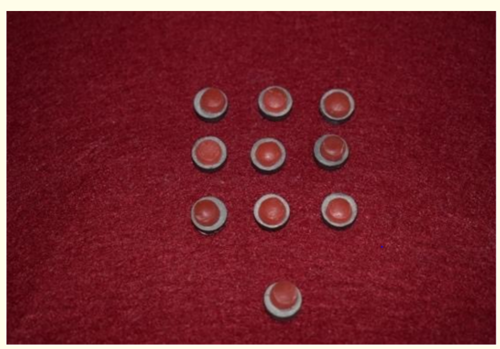


Figure 4: *The samples after adding wax discs.*

The following primers were applied on 30 discs Alloy Primer (AP) and Z-Prime Plus. All primers were applied in a uniform coating with a brush and were allowed to air dry, according to the manufacturer's instructions. Acrylic resins were applied after 5 minutes.

Denture base heat-polymerized acrylic resin (HPAR) (Meliodent Heat Cure, GERMANY) was mixed and placed into the molds according to the manufacturer's instruction. A trial pack was completed, and excess material was removed from the opened flask. Then second pack was completed and held for 5 minutes.

Figures 5, 6: *Dental plaster poured into the dental flasks.*

The acrylic resin was heat polymerized for 60 minutes, and the polymerization began in water bath at room temperature, then heated up to 100°C over half an hour, the flask was held at this temperature for 30 minutes and then left to cool down at room temperature.

Figure 7: *The shape of the specimens after wax melted.*

Figure 8: *Packing of heat cure acrylic resin.*

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

Figure 9: *The final shape of samples after finishing.*

All the samples were embedded in the self-cure acrylic resin (Imicryl, TURKEY) by using metallic mold (12 mm in thickness and 20 mm in diameter).

Shear bond strength was measured by using the universal testing machine (Instron 3345 model, USA) with a crosshead speed of 0.5 mm/min.

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 de bonding shear bond strength converted into MPa.

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

Statistical calculation was performed with (Number Cruncher Statistical System) 2007 Statistical Software (Utah, USA) program for Windows. The data were analyzed with one-way ANOVA to compare groups, Post Hoc Tukey multiple comparison test was utilized in the comparison of two groups. Statistical significance level was established at $P < 0.05$.

Result

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).

	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

Table 2: *Mean shear bond strength for all metal alloys groups (MPa).*

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

Table 3

	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

Table 4: *Shear Bond Strength means for Primers groups (MPa).*

The Shear Bond Strength of the Z Prime Plus group was statistically significantly higher than the Shear Bond Strength of alloy Primer.

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

Table 5: *Two-way analysis of variance for Shear Bond Strength.*
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).*

	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

Table 6: *Shear Bond Strength of Alloy Primers.*

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).

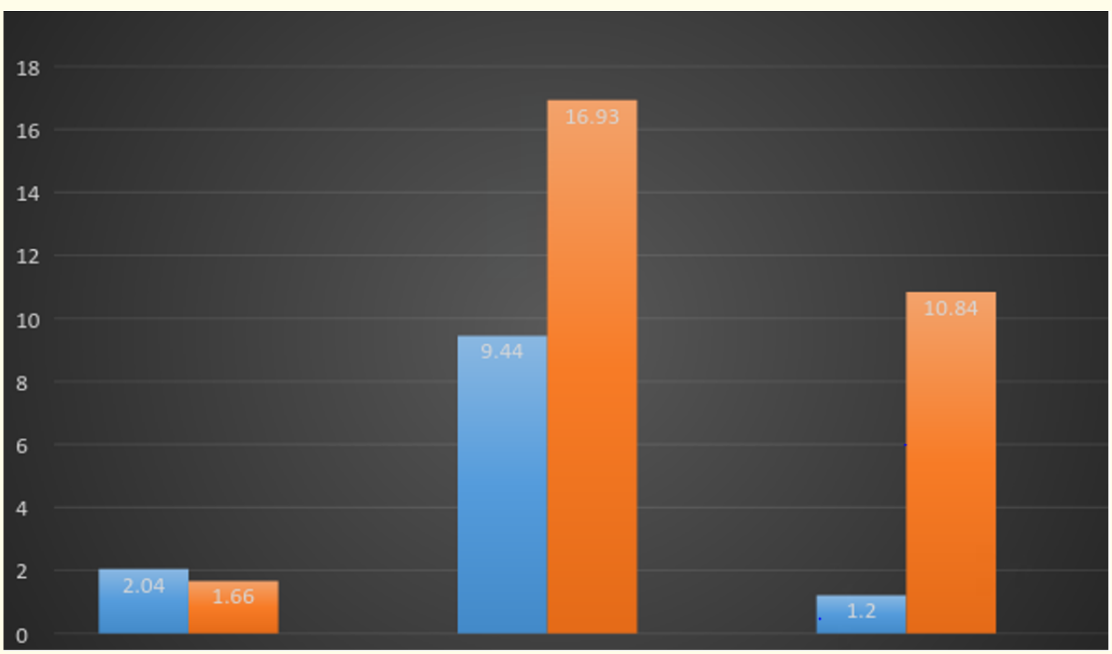


Figure 10: *The effect of Alloy Primer and Z Prime Plus on the Shear Bond Strength of Cobalt, Nickel and Titanium alloys.*

Discussion

Removable partial denture is considered as a good treatment option for patients not only because it is more economical than tooth or implant retained bridges. But also, because they can replace missing oral structures [8]. The common base metal alloys that are used in RPD fabrication are cobalt-chromium, nickel-chromium and titanium alloy.

The bonding between the heat cured acrylic resin and the metal alloys have been improved using surface treatments. These procedures include sandblasting to roughen the surface for micromechanical retention and/ or chemical etching [13] or electrolytic etching [14].

A failure of the bond between acrylic resin and metal can result in the accumulation of stain/discolouration harbouring of microorganism and the delamination of the resin and the prosthetic teeth from the framework [4].

Titanium is a new material introduced into RPD construction because it has excellent biocompatibility, corrosion resistance and good strength.

The desirable physical and mechanical properties of the Ti-6Al-4V and commercially pure titanium (CP) are very similar to each other [4]. Clinical report of the use commercially pure titanium [15] for denture framework highlighted at the junction between resin and metal due to stain penetration, despite using adhesive resin that contained 4-META (META-DENT, Sun Medical, Kyoto, Japan) [4].

Further investigation showed that the shear bond strength of the resin containing 4-META to pure titanium decreased after thermocycling [16]. Usually there is a stable titanium oxide layer over the surface of pure titanium metal. The metal primer will adhere to this layer rather than adhere to CP titanium itself, and the chemical bond can result from the reaction of metal oxide with phosphoric acid or carboxylic acid derivatives [4]. The CP titanium framework being more flexible than the cobalt-chromium alloy in function may cause the resin debonding.

It has been reported that sandblasting removes the contaminated layer and improved the wettability of the alloy [17], while increasing the surface area creating a roughened surface conductive for mechanical interlocking [19]. Metal sandblasting with Al_2o_3 particles produce a passive film containing Ni, Co, Cr oxides. [18]. Giachetti., *et al.* [20] used scanning electron microscopy (SEM) to make a morphological analysis of sandblasted metal surface with different sizes of alumina particles (50μ vs 150 μm). The result showed that the surface treated with 150 μm alumina particles presented deep and large cavities where the resin can penetrate more compared to the surface treated with 50 μm which appeared irregular and rough.

However, ultrasonic cleaning of sandblasted surface to remove loose particles should be avoided as it can decrease the adhesive strength of the luting resin [21-23].

To get a good result of this technique there are some parameters in the airborne 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 [24,26].

The use of alloy primers that contains VBATDT and MDP as an active group has been shown to increase the bond strength between base metal alloys and acrylic resin [25]. MDP has an ester phosphate group which provides a good chemical bond the metal oxide (Cr_2O_3) layers that covered the surface of the cobalt-chrome [27]. Mercapto groups in the VBATDT monomer react with the noble metal alloys and produce a chemical bond between the metal-resin interfaces.

Although using alloy primer is a simple and a good way to increase the durability of the shear bond strength between the acrylic resin and metal alloys, this bond depends on the composition of the metal alloy [28,29].

Thus, meta fast has been suggested or titanium alloys, while Metal Primer and Alloy primer has been recommended for use with base metal and noble alloys.

Use of alloy primers has thus been shown to increase the durability of clinical bond strength of the resin to RPD framework compared to non-primed framework [30]. 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 [33,34] storage in sodium hypochlorite (NaClO) resulted in lower bond strength [35]. In our study we choose distilled water as storage media before the test. To stimulate clinical condition thermocycling was used. Mair found that oral cavity temperature changes from -4^{o}C to 0^{o}C when eating ice cream and between 60^{o}C - 65^{o}C when eating a hot cheese sandwich [36]. As most of the time dental restorations are subjected to little temperature variation, thermocycling may not have a significant effect but it can lead to spontaneous debonding of specimens [35].

A short regimen of thermal cycling is recommended by ISO TR 11450 standard (2003) is 500 cycles [36]. According to the previous studies the number of cycles was decided 6000 thermal cycles are equal to 5 years of clinical use [37,38]. So, 5000 cycles that is used in our study are equal to 4 years of clinical function. Celik., *et al* [39]. 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 [39]. Most of *in vitro* studies used monotonic tests as compression, shear or tensile strength to examine the mechanical properties of the dental materials [42]. 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 [41]. Oilo [42] classified the bond strength test into quantitative tests and qualitative tests. The quantitative tests study predicts the lifetime of the bond and the load capacity and qualitative tests study bond failures [42].

In the static tests, force applied when the specimen is in stationary 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 < 3 mm^2 bond area and macro tests with > 3 mm^2 [43]. The macro bond strength can be measured in tensile, shear or using a push-out protocol [44]. 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 [45]. 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 [46-50]. 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. [42]. 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 demonstrates better bond strength than do the primers contain 4-META monomer [18]. 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., *et al* [30].

Sandeep., *et al*. investigated the effect of sandblasting and metal primer on the SBS between Co-Cr alloy and heat cure acrylic resin [13]. 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* [20]. 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.

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. However, Kawaguchi T., *el al.* results for Co-Cr was opposite to our result, it was 21.6 ± 3.7 MPa.

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 when 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.

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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