

Shear Bond Strength of Metal Orthodontic Brackets on Prepared Silver–Palladium Alloy Surfaces; An *In Vitro* Study

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ABSTRACT

Orthodontists are facing the problem of bonding orthodontic brackets to non-enamel surfaces, especially in full metal crown restorations. Limited information is available about the strength of bond of orthodontic attachment to dental based alloy. This *in vitro* study aimed to evaluate the effects of three metal primers on the shear bond strength of orthodontic brackets bonded to sandblasted silver–palladium alloy with light-activated orthodontic adhesive. Forty silver–palladium specimens were sandblasted and assigned to four groups: (1) No metal primer, (2) Reliance Metal Primer, (3) ML Primer and (4) Alloy Primer. The brackets were bonded to the conditioned alloy using the light-activated orthodontic resin (Transbond™ XT). After being incubated in deionized water at 37° C for 24 hours, thermocycling (5,000 cycles) was performed. Shear bond strength was measured with a universal testing machine at 1 mm/min. The data were analyzed with one-way ANOVA and the Adhesive remnant index was assessed. Scanning electron microscopy were assessed on intact, sandblasted and after de-bonding specimens. The results showed that the mean shear bond strength values (MPa) for no metal primer, Reliance metal primer, ML primer and Alloy primer were 9.19 ± 3.39 , 15.00 ± 3.10 , 11.37 ± 2.84 and 16.30 ± 3.63 respectively, while the trends of Adhesive remnant index (more than 50%) in each group were 1, 0, 2 and 3, respectively. The shear bond strengths in Reliance Metal Primer and Alloy Primer groups were significantly higher than no primer and ML primer groups. ($p < 0.05$) With this *in vitro* study, sandblasting with/without metal primer yielded adequate shear bond strength (>5 MPa) of metal brackets on silver–palladium alloy with light-activated adhesive resin. Reliance Metal Primer would be the appropriate metal primer in this study because of the high shear bond strength and the low ARI scores.

Keywords : Metal primer/ Orthodontic bracket/ Shear bond strength/ Silver–palladium alloy

Introduction

The technique of bonding orthodontic metal brackets to enamel surfaces with adhesive resins has been developed to the extent that the strength of the bond is satisfactory.¹ Nevertheless, orthodontists are facing the problem of bonding orthodontic brackets to non-enamel surfaces, one example is in full-coverage metal crown restorations. Silver–palladium-based alloys are one of the materials commonly used in full metal crown restorations. Their corrosion properties, malleability and marginal adaptation are good and they cost less than high noble metals.^{2,3}

The adequate shear bond strength of the adhesive resin–metal alloy interface is needed for the success of the orthodontic bracket bonding procedure. The strength of the bond between adhesive resin and metal is inferior to that between adhesive resin and enamel/porcelain, resulting in its bond failure. Initially, bond strength depends on mechanical retention only. After the development of the chemical agent (also known as metal primer) was introduced, the strength of the bond between alloy and adhesive resin was then improved. Nowadays, there are many commercial metal primers available according to the functional molecules which can condition the metal alloy differently. It has been demonstrated, *in vitro*, that metal primers effectively

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enhance the bonding capacity of the adhesive resin to noble alloys, especially in gold alloys.^{4,5} However, a limited information is available about the strength of bonds to silver palladium based alloy. The purpose of this study was to evaluate the effect of three different metal primers on the shear strength of the bond between metal orthodontic brackets and silver palladium alloys with light-activated orthodontic adhesive. The hypothesis was there would be no difference in the shear strength of bond between the orthodontic bracket lined with adhesive resin and the silver palladium alloy prepared with three commercial metal primers.

Materials and methods

Forty silver-palladium-based alloys (AURO W-LITE; AURIUM Research, San Diego, CA, USA) were cast in square-shaped specimens with the dimensions of 6x6x1.5 mm. Each alloy specimen was embedded in a self-cured acrylic block (Jet™ Tray Resin, Lang Dental, Wheeling, IL, USA) and the alloy surface was oriented parallel to the force direction during the shear test. All specimens were polished in the laboratory and re-polished by the researcher with a silicon carbide paper in a polishing machine (MOPAO 160E, MEGA

Advance, HK, China) in a sequence of 1,500 and 2,000 grid sizes and then polished with a buffing cloth for 30 seconds to achieve a glazed surface. This procedure was to prepare a flattened and standardized surface as a baseline finish.

All specimens were air-abraded using 50 µm Al₂O₃ particles (DANVILLE, San Ramon, CA, USA) for five seconds at a 90-degree angle and a five mm nozzle-metal surface distance. The specimens were cleaned in an ultrasonic bath (UC125H BioSonic®, Coltene/Whaledent, Altstätten, Switzerland) filled with deionized water for five minutes and dried using an oil-free stream. The specimens were randomly divided into four groups. The first group served as a control group (C), where the specimens were sandblasted but unprimed. In the remaining three groups, the following primers were applied: Reliance Metal Primer (RM), ML Primer (ML), and Alloy Primer (AP). A microbrush was used to apply the primer in each group in a uniform coating and the primers were allowed to air dry, strictly according to the manufacturers' instructions. Information on the principal materials used in this study is summarized in Table 1.

Table 1 Dental alloy, Metal primers and Adhesive system used in this study

Material	Manufacturer	Functional Group	Component	Batch number
Dental alloy				
AURO W-LITE	AURIUM Research, San Diego, CA, USA	-	Ag 75%, Pd 19.97%, In 3%, Zn 2%	7222070
Metal primers				
Reliance Metal Primer (RM)	Reliance Orthodontic Products, Inc., Itasca, IL, USA	Carboxyl	4 META	141390
ML Primer (ML)	SHOFU, Kyoto, Japan	Phosphate, Thione	6 MHPA, 10-MDDT	051260
Alloy Primer (AP)	Kuraray, Osaka, Japan	Phosphate, Thiol	MDP, VBATDT	00444C
Adhesive system				
Transbond™ XT Primer	3M Unitek, Monrovia, CA, USA	-	BisGMA, Silane, Dimethacrylate	N513350
Transbond™ XT light Cure Adhesive	3M Unitek, Monrovia, CA, USA	-	Dimethacrylate	N494631

The maxillary right central incisor bracket base (GEMINI MBT 0.022 Twin; 3M Unitek, Monrovia, CA, USA) was applied with Transbond™ XT light cure adhesive (3M Unitek, Monrovia, CA, USA). The brackets with adhesive were placed at the center of the alloy surface. Each bracket was subject to a 200g compressive force with a force gauge (CORREX D-7530, Dentaaurum, Pforzheim, Germany). The excessive bonding resin was removed with a scaler. Then, all brackets were light cured using a DEMI LED curing unit (Kerr Corporation, Middleton, WI, USA) for 20 seconds on each side (mesial, distal, incisal, cervical edge). A minimum light intensity of 1,100 mW/cm² was verified by the unit's built-in curing radiometer.

According to ISO 10477,⁶ after bonding, all samples were stored in deionized water and kept in an incubator (Mettmert BE-400, GmbH. Co. KG, Burladingen, Germany) at 37°C for 24 hours. Thermocycling was performed between 5°C and 55°C for 5,000 cycles (30 seconds in each bath with a dwell time of ten seconds) (Thermocycling machine, HWB332R, CWB332R, TC301, King Mongkut's Institute of Technology, Bangkok, Thailand). After this aging process, the brackets were de-bonded using a universal testing machine (Instron 5566, Instron Calibration Laboratory, Canton, MA, USA). For the shear mode, the applied force must be located as close to the interface as possible and parallel to the alloy surface. The crosshead speed was set at 1 mm/min and 10,000 g of load cell was used. Calculated shear bond strength was determined by dividing the recorded force at which bond failure occurred (in Newtons) by the bonding area (in mm²). The average surface area of the GEMINI MBT bracket base was 10.9667 mm².

After de-bonding of the brackets, images of the bracket bases were recorded using a digital single lens reflex camera (Canon EOS 550D with EF-S 60 mm. f/2.8 Macro USM, Oita, Japan). The distance between bracket base and camera lens was the same for each

specimen and the camera was set to a 1:1 magnification value. The areas of bare surface on the de-bonded brackets were calculated using the ImageJ program (Wayne Rasband, Public domain) and converted to a residual adhesive score. Remaining adhesive was recorded as a Adhesive remnant index (ARI) score from 0 to 3. Scores were modified according to Artun and Bergland⁷, score 0 means that there was no adhesive left on the alloy surface. Score 1 means that there was less than 50% of the adhesive left on the alloy surface. Score 2 means that there was more than 50% of the adhesive left on the alloy surface. Lastly, score 3 means that the entire adhesive was left on the alloy surface.

A scanning electron microscope (JEOL JSM-5910LV, JEOL Ltd., Tokyo, Japan) was used with x200 and x2000 magnification, to examine some specimens: after polishing with a buffing cloth, after sandblasting, and after de-bonding.

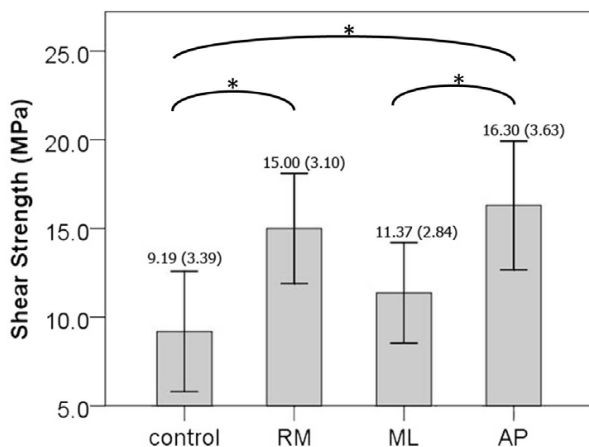
Descriptive statistics, including the mean, standard deviation, minimum and maximum values were calculated for each of the four tested groups. The analysis of variance was used to determine whether significant differences existed between the groups. If a significant difference was present, a Tukey's Post Hoc test was used to identify which of the groups were different. The percentage of the ARI scores was also calculated.

Results

The mean shear bond strength results are presented in Table 2. The data satisfied the normality of distribution and homogeneity of variance assumptions; the parametric statistical tests were applied. The obtained data were statistically calculated through ANOVA and Tukey's Post Hoc test, both of which revealed significant differences ($p < 0.05$) among the groups as shown in Figure 1.

Table 2 Mean shear bond strength, standard deviation and range of values (MPa)

Group	N	Mean	S.D.	Minimum	Maximum
Control	10	9.19	3.39	4.01	13.55
Reliance metal primer	10	15.00	3.10	10.15	20.85
ML primer	10	11.37	2.84	7.89	15.64
Alloy primer	10	16.30	3.63	10.91	22.14

**Figure 1** Histogram of the Means and Standard deviation values (MPa)

* = The statistical differences were found among the groups ($p < 0.05$)

The shear bond strengths were significantly higher in two groups, namely, Reliance Metal Primer (15.00 ± 3.10 MPa) and Alloy Primer (16.30 ± 3.63 MPa), whereas no primer (9.19 ± 3.39 MPa) and ML Primer (11.37 ± 2.84 MPa) showed lower results. However, the Reliance Metal Primer and Alloy Primer means were not found to be statistically different.

The frequency distributions of the ARI scores of each group are presented in Table 3.

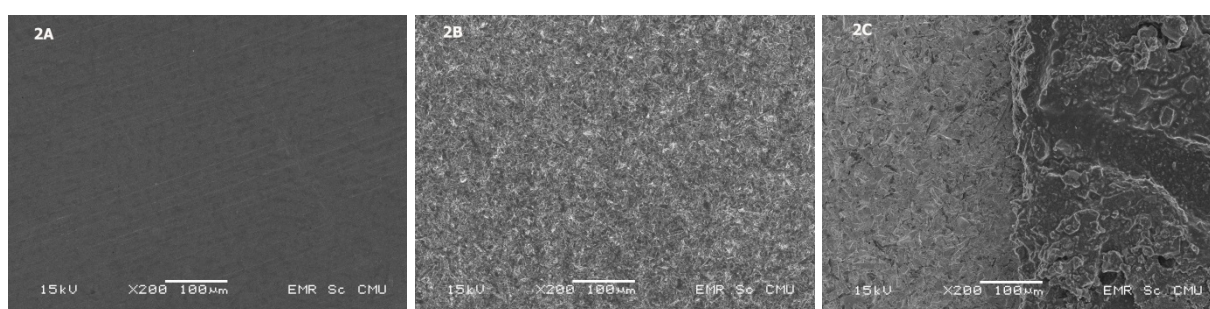
With the use of Reliance Metal Primer, there was a highest percentage (70%) of ARI scores of 0, which indicated all of the adhesive resin was on the bracket.

For no primer and ML Primer groups, most of the specimens displayed ARI scores of 1 or 2, suggesting a trend to display that adhesive remnant was on both the bracket and alloy sides of the fracture. The ARI score of 3 was mostly found in the Alloy primer group, which showed that all of the adhesive resin was on the alloy surface.

SEM images were used to examine the characteristics of the silver palladium surfaces, after undergoing polishing procedures and sandblasting. Figure 2A represents the SEM image with the 200x magnification of the polished alloy showing a smooth surface and with a small number of scratches, while a remarkably rough surface and irregular structure with sharp-edge projections across the surface was found on the sandblasted specimen as in Figure 2B. Some SEM images with the 200x magnification of alloy surfaces after bracket de-bonding were selected and shown in Figure 2C. The irregular and rough surface of alloy on the side with no adhesive remaining was similar to Figure 2B, whereas the adhesive resin was found on the other side with some imprints of the bracket base. These combination findings are commonly found in the specimens with ARI Scores of 1 and 2 under the bonding area.

Table 3 The frequency distribution of the ARI scores in each group

ARI scores Group	0	1	2	3	Total
Control	4(40%)	5(50%)	1(10%)	0(0%)	10(100%)
Reliance Metal Primer	7(70%)	1(10%)	1(10%)	1(10%)	10(100%)
ML Primer	1(10%)	2(20%)	5(50%)	2(20%)	10(100%)
Alloy Primer	0(0%)	1(10%)	4(40%)	5(50%)	10(100%)

**Figure 2** The SEM image (200x magnification) of the polished alloy (Figure 2A), the image of the sandblasted alloy before bonding (Figure 2B) and The SEM images (2000x magnification) of the alloy surfaces after bracket debonding (Figure 2C).

Discussion

The main purpose of this study was to evaluate the effect of three metal primers on shear bond strength of metal orthodontic brackets on silver-palladium alloys with light-activated orthodontic adhesive. Reynolds⁸ suggested that minimum bond strength of 5–8 MPa was adequate for orthodontic bonding. In this *in vitro* study, the lowest means of shear strength values were greater than 8 MPa. It was also noted that the sandblasting procedure without metal primer provided adequate shear strength of the bond. However the *in vitro* testing of bond strength may not closely simulate the oral environment, which is influenced by numerous factors, including contamination with saliva or blood and degradation of the adhesive when exposed to saliva and temperature variations caused by food or drinks.⁹ Therefore, the results of an *in vitro* study may show higher shear bond strength than what really prevails in a clinical situation.

The authors tried to explore the effective and efficient methods for bracket bonding to a silver-palladium alloy surface using sandblasting and metal primers. The results suggest that the use of metal primer combined with the sandblasting procedure effectively enhances the bonding capacity of the adhesive resin to silver palladium alloys. So, as a result of this *in vitro* findings, Alloy Primer and Reliance Metal Primer showed the superior bond strength.

Several *in vitro* studies indicated that, when metal primers containing 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithione (VBATDT) are applied on noble alloys, the shear bond strength was significantly increased.^{10,11} Furthermore, previous studies found that the metal primer containing carboxyl or phosphoric acid derivatives provided strong adhesion to base alloys.^{12–15}

In this study, Alloy Primer exhibited the highest shear strength of the bond when compared to other metal primers because it contains two functional monomers, VBATDT and 10-methacryloyloxydecyl dihydrogen phosphate (MDP). VBATDT is a chemical agent in the thiol group in metal primer and MDP contains phosphoric acid as a functional group. It can be inferred that VBATDT in Alloy Primer had an effect on a noble palladium, whereas MDP bonded to silver, indium and zinc that were the base components in the specimens. However, Reliance Metal Primer with only one functional monomer (carboxyl group) provided a nonsignificant difference in bond strength with the Alloy primer group. Moreover, ML primer also contains two functional groups, 6-Methacryloyloxyhexyl phosphonoacetate (6MHPA) as a phosphoric acid group and 10-Methacryloyloxydecyl-6, 6-Dithiooctanoate (10-MDDT) as a thione group. Obviously the shear bond strength of ML group was significantly lower than Alloy primer. Sanohkan S. et al.¹⁶ reported that shear bond strength of ML primer was significant lower than Alloy primer when bonded to cobalt chromium alloy and acrylic resin. Their hypothesis was that 6MHPA in ML primer had a shorter hydrocarbon chain than MDP in Alloy primer. So, MDP in alloy primer has more hydrophobicity and more flexibility than 6MHPA in ML primer.

Another important factor was the ARI scores which were considered after debonding procedure. In orthodontics, the location of bond failure is significant because an effort is made to maintain a sound restoration surface or tooth surface after the de-bonding process. Thus, in order to minimize the process of re-polishing the full metal restoration, it is more desirable for bond failure to occur at the alloy–adhesive interface (ARI 0 or 1) than within the adhesive or at the adhesive–bracket junction (ARI 2 or 3). Furthermore, a smaller adhesive remnant means less chair time for removal of the

adhesive and less risk of scratches of alloy surface during polishing procedures. The ideal bonding system in orthodontics should leave a smooth alloy surface without large amounts of adhesive to remove. On the other hand, when considering the superior bonding characteristic on alloy surface, bond failure should occur more at the adhesive–bracket junction proving that the bond between the alloy surface and the adhesive is better than the bond between the adhesive and bracket base. Table 3 shows that Reliance Metal Primer exhibited mostly ARI scores of 0, meaning no adhesive left on the alloy surface and also provided comparable bond strength with Alloy primer group. However, the rough and irregular surfaces created by sandblasting procedures remain as seen from SEM images (Figure 2C) and thus require the re-polishing procedure. The other groups, ML Primer and Alloy Primer mostly had adhesive resin on the alloy surface showing the better bond between alloy surface and the adhesive. So, there would be a greater chance of damaging the alloy surface and of using more chair time to remove the adhesive remnants when ML Primer or Alloy Primer is used. Therefore, Reliance Metal Primer exhibited superior behavior in term of ARI scores when consider the chairtime of debonding. However, there was not much different in bond strength between Reliance primer group and Alloy primer group. If the maximum bond strength is needed, the high ARI scores should occur along with the increased bond strength.

The results of this *in vitro* study suggest that among four approaches, Reliance Metal Primer is the appropriate metal primer because of the high shear bond strength and the lowest ARI scores for convenient debonding procedure. Both Reliance Metal Primer and Alloy Primer groups showed significantly higher shear bond strength than did ML Primer and sandblasting treatment groups. However, most Alloy Primer specimens produced ARI scores of 3, whereas the Reliance group produced ARI score of mostly 0.

According to the study designs, the type of alloy chosen in this study was silver–palladium and there is quite limited information about bond strength when it is bonded to metal orthodontic brackets. The natural crowns have a horizontally curved surface. Curved orthodontic bracket bases follow the surface of the crowns and thus offer the best fit. In this study, the alloy specimens were cast with flat surfaces because of the standardization of polishing procedure which followed the protocol of ISO⁶ and other international studies. However, the authors recognize that the shear strength of the bond might not be similar to that in the clinical situation. Different researchers and testing designs reported different shear bond strength values. Because of the lack of standardization, many *in vitro* studies which have been published can only be evaluated individually.^{17,18} So, this study was designed according to the recommendations of ISO 10477:2004.⁶ The number of samples in each group which recommended by ISO was five. In this study, ten metal plates were prepared. In addition, a polymer-based crown and bridge material would pass the requirements of ISO if test results show that at least 4 out of 5 specimens demonstrate shear bond strength equal or greater than 5 MPa. In this study, the shear bond strength of every specimen primed with Reliance Metal Primer, ML Primer and Alloy Primer was greater than 5 MPa. In the group with no metal primer, 2 of 10 specimens yielded shear bond strength of less than 5 MPa (4.01 and 4.54 MPa respectively), but the group still passed the requirements of ISO. To summarize, all of the groups achieved the requirements of ISO 10477:2004.

Finally, additional research is needed to determine if the use of metal primer without sandblasting could yield acceptable shear bond strength. In the clinical situation, the dental practitioner may not know what exactly the material of the metal crown was fabricated. It would be advantages if the suitable metal primer or simple method would be clarified in bonding with all dental alloys.

Conclusion

It can be concluded from this *in vitro* study that sandblasting with/without metal primer yielded adequate shear strength in the bond between silver–palladium alloy and adhesive resin when bonded to metal orthodontic brackets. Reliance Metal Primer would be the appropriate metal primer in this study because of the high shear bond strength and the lowest ARI scores.

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ความแข็งแรงของการยึดติดแบบเนียนของแบร็กเกตจัดฟันโลหะบนโลหะผสมเงินพัลลาเดียมที่ถูกเตรียมผิว; การศึกษานอกกาย

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บทคัดย่อ

การยึดติดเครื่องมือจัดฟันบนพื้นผิวที่ไม่ใช่เคลือบฟันเป็นปัญหาอย่างต่อเนื่องของทันตแพทย์จัดฟัน โดยเฉพาะพื้นผิวของครอบฟันโลหะเต็มชิ้นในฟันหลัง ข้อมูลวิธีการที่เหมาะสมสำหรับการยึดติดกับพื้นผิวดังกล่าวให้มีประสิทธิภาพและคงอยู่ตลอดการรักษาจำเป็นจำกัด การศึกษานอกกายนี้มีวัตถุประสงค์เพื่อศึกษาถึงผลของเมทัลไพโรเมอร์ 3 ชนิดต่อความแข็งแรงของการยึดติดแบบเนียนของแบร็กเกตจัดฟันโลหะกับโลหะผสมเงินพัลลาเดียมที่ถูกแปดด้วยทรายด้วยวัสดุยึดติดที่บ่มด้วยแสง เตรียมชิ้นงานทดลองโลหะผสมเงินพัลลาเดียมจำนวน 40 ชิ้น นำชิ้นงานมาแปดด้วยทรายแล้วแบ่งชิ้นงานเป็น 4 กลุ่มดังนี้ กลุ่มที่ 1 ไม่ใช้เมทัลไพโรเมอร์ กลุ่มที่ 2 ใช้เมทัลไพโรเมอร์ไลแอนซ์ กลุ่มที่ 3 ใช้เมทัลไพโรเมอร์เอ็มแอล และกลุ่มที่ 4 ใช้เมทัลไพโรเมอร์อัลลอย นำแบร็กเกตจัดฟันโลหะมายึดติดชิ้นงานด้วยวัสดุยึดติดที่บ่มด้วยแสง (ทรานส์บอนด์เอ็กซ์) แล้วนำไปเก็บบ่มในน้ำไร้ไอออนที่อุณหภูมิ 37 องศาเซลเซียส 24 ชั่วโมง จากนั้นนำไปผ่านกระบวนการเทอร์โมไซคลิงจำนวน 5,000 รอบ นำชิ้นงานมาหาค่าความแข็งแรงของการยึดติดแบบเนียนด้วยเครื่องทดสอบเอนกประสงค์ ความเร็วหัวกด 1 มิลลิเมตรต่อนาที และบันทึกดัชนีของวัสดุยึดติดที่เหลือหลังการทดสอบ นำข้อมูลค่าความแข็งแรงของการยึดติดมาวิเคราะห์ทางสถิติโดยใช้การวิเคราะห์ความแปรปรวนทางเดียว นำชิ้นงานก่อนทดสอบ ชิ้นงานถูกแปดด้วยทราย และชิ้นงานหลังการทดสอบบางอย่างถูกนำไปศึกษาพื้นผิวด้วยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด ผลการศึกษาพบว่าค่าเฉลี่ยความแข็งแรงของการยึดติดแบบเนียนและส่วนเบี่ยงเบนมาตรฐาน และค่าแนวโน้มดัชนีของวัสดุยึดติดที่เหลือที่มากกว่าร้อยละ 50 มีดังนี้ กลุ่มที่ 1 ไม่ใช้เมทัลไพโรเมอร์ 9.19 ± 3.39 เมกาปาสคาล ดัชนี 1 กลุ่มที่ 2 ใช้เมทัลไพโรเมอร์ไลแอนซ์ 15.00 ± 3.10 เมกาปาสคาล ดัชนี 0 กลุ่มที่ 3 ใช้เมทัลไพโรเมอร์เอ็มแอล 11.37 ± 2.84 เมกาปาสคาล ดัชนี 2 และกลุ่มที่ 4 ใช้เมทัลไพโรเมอร์อัลลอย 16.30 ± 3.63 เมกาปาสคาล ดัชนี 3 ค่าเฉลี่ยความแข็งแรงของการยึดติดแบบเนียนในกลุ่มเมทัลไพโรเมอร์ไลแอนซ์และกลุ่มเมทัลไพโรเมอร์อัลลอยมีค่าสูงกว่ากลุ่มไม่ใช้เมทัลไพโรเมอร์และเมทัลไพโรเมอร์เอ็มแอลอย่างมีนัยสำคัญทางสถิติ จากการศึกษาการเตรียมพื้นผิวโลหะด้วยการแปดด้วยทรายและการใช้หรือไม่ใช้เมทัลไพโรเมอร์ให้ค่าความแข็งแรงของการยึดติดแบบเนียนของแบร็กเกตจัดฟันโลหะกับผิวโลหะผสมเงินพัลลาเดียมด้วยวัสดุยึดติดที่บ่มด้วยแสงที่เพียงพอ (มากกว่า 5 เมกาปาสคาล) นอกจากนี้เมทัลไพโรเมอร์ไลแอนซ์มีความเหมาะสมในการศึกษานี้เนื่องจากให้ค่าความแข็งแรงของการยึดติดแบบเนียนสูงและให้ค่าดัชนีของวัสดุยึดติดที่เหลือต่ำ

คำไวยุทธ: เมทัลไพโรเมอร์/ แบร็กเกตจัดฟัน/ ความแข็งแรงของการยึดติดแบบเนียน/ โลหะผสมเงินพัลลาเดียม

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