Eur. J. Prosthodont. Rest. Dent., Vol.19, No. 1, pp 43-48
Printed in Great Britain

doi:10.1922/EJPRD_935Newsumount06

An Investigation into the Effects of Metal Primer and Surface Topography on the Tensile Bond Strength Between Cobalt Chromium Alloy and Composite Resin

David Newsum*, Andrzej Juszczyk†, Robert K.F. Clark‡ and David R Radford§

Abstract - *This study examined the influence of surface preparation and metal primer on the tensile bond strength between cobalt chromium alloy and composite resin. The bond strength between 168 cobalt chromium alloy dumb-bells with one of three test surfaces (beaded, machined or sandblasted) to composite resin were tested. Half of each group were treated with metal primer. The weakest bond strength was produced by the unprimed machined surface, many specimens failing before testing. The metal primer increased the bond strengths of all groups tested. The greatest bond strengths were achieved with the primed beaded and sandblasted surfaces. Within the limits of the study it has been shown that the surface preparation of the cobalt-chromium alloy did influence tensile bond strengths with composite resin and Metal Primer II increased the tensile bond strengths for all groups tested. The sandblasted surface treated with Metal Primer II is recommended for the bonding of composite resin to cobalt chromium alloy.*

KEY WORDS: primer, composite resin, surface, cobalt chromium, bond strength

INTRODUCTION

Modern laboratory composite resins offer aesthetics comparable to dental porcelains and so if these can be successfully combined with cobalt chromium alloy frameworks, overlay dentures become a more viable option for many patients. There has however, been little research published on bond strengths between composite resin and cobalt chromium alloy.

Numerous investigators have investigated the bond strengths of metals and restorative materials. The first reports of a chemical bond were reported by Tanaka *et al*[1] with the use of 4-META (4-methacryloxyethyl trimellitate anhydride) and thermosetting acrylic resin with a nickel chromium alloy. It has also been reported that a chemical bond exists between cobalt-chromium alloy and acrylic resin when utilising 4-META[2]. Primers such as 4-META have also been shown to influence bond strengths with other materials such as composite resin[3] where tensile bond strengths of 9.5 MPa were attained, which was a significant improvement on mechanical retention (6.9 MPa).

It has been demonstrated that cobalt-chromium alloy achieves greater shear bond strengths (37.3 MPa) with 4-META than nickel-chromium alloy (31.6 MPa)[4]. In a study that compared two primers, 4-META and 10-methacryloxydecyldihydrogen phosphate (MDP), with cobalt-chromium alloy, MDP produced peak shear bond strengths of 44.0 MPa compared to 4-META which produced peak values of 34.1 MPa. Later studies again confirmed the bond strength produced by MDP to be superior for a variety of manufacturers' composite resin materials. Kern and Thompson confirmed MDP to provide resin alloy bonds with shear bond strengths reported of up to 53.7 MPa, greater than all other systems tested.

In a direct comparison study MDP and MEPS in MMA (thiophosphate methacryloxyalkyl derivatives in methyl methacrylate) were investigated. Specimens with no primer produced shear bond strengths four times that of early studies[3]. MDP primer recorded the highest shear bond strength (67.1 MPa) after thermocyling. The MEPS MMA primer produced varied results with the resin systems ranging from 32 to 70.4 MPa.

Orchard *et al*[9] reported similar results with shear bond strengths of MDP primer and an unspecified methacrylic primer. The results showed that MDP primer was superior to its methacrylate counterpart although the bond strengths reported are significantly lower than those reported by other authors [5,6,7,8].

Most recently Behr *et al*[10] tested shear bond strengths of composite resin with different surface preparations on cobalt-chromium alloy. They investigated silicoating, an experimental new titanium coating and the use of adhesive primers. They concluded that functional monomers such as Metal Primer II were the treatment of choice for bonding cobalt chromium alloy and composite resin.

There has been extensive work on retaining acrylic resin mechanically and adhesively. It has been shown when bonding acrylic resin to titanium alloy that sandblasting or silicoating the titanium surface produced greater bond strengths[11]. Tanaka *et al*[2] postulated, and this was later

---

* BDS, MFD RCSI, MSc, MRCPS, MRD RCSEd
† LCGI, PhD
‡ BDS, PhD, FDS
§ BDS, PhD, FDS RCS, MRD

confirmed by Salonga *et al*[12], that the difference in bond strengths observed between nickel chromium and cobalt chromium with various substrates was attributed to the greater oxide layer produced on cobalt chromium, in part due to the greater percentage of chromium.

Retentive shapes altering surface contour have long been incorporated into castings to increase retention and surface area. Barzilay *et al*[3] reported on the influence of retention beads and the shear bond strengths of composite resin. They demonstrated that smaller retention beads (350µm) produced a statistically significant improvement over larger retention beads used (430µm).

The aim of this study was therefore to examine if the surface treatment and surface contour of cobalt chromium alloy and the application of a metal primer influenced the tensile bond strength between the cobalt chromium alloy and composite resin.

MATERIALS AND METHODS

Experimental Design

84 test specimens were produced and divided into six test groups. The test groups comprised three different test surfaces: beaded, machined and sandblasted and with or without bonding agent. The six groups were 1 Beaded, 2 Beaded with primer, 3 Machined, 4 Machined with primer, 5 Sandblasted, 6 Sandblasted with primer. A test specimen consisted of two cobalt chromium alloy dumbbells bonded together at the test surfaces with laboratory composite resin. Test specimens were stored for seven days in water at 37°C in a thermostatically controlled incubator before testing. The test specimens were placed in the Instrom 1193 Universal testing machine and loaded to failure in tension, with a crosshead speed of 1mm per minute with a 2000N load cell. The behaviour of the tested specimens under load and the final load failure was recorded by means of a chart recorder unit with a paper speed of 0.5mm/sec.

Alloy dumbbell production

Cast acrylic rods 6mm in diameter were machine turned on a lathe to produce uniform dumbbell shaped acrylic patterns. One hundred and sixty-eight identical acrylic patterns were produced and invested and cast in cobalt chromium alloy. The resulting alloy dumbbells were sandblasted and electro brightened.

Surface preparation

The test surfaces were prepared as follows:

Beaded surface: A beaded surface was produced on the acrylic patterns before investing by adhering 0.5mm beads to the pattern surface ensuring maximum coverage. Once cast and divested the beaded test surface was sandblasted using the same protocol as the sandblasted surface protocol.

Machined Surface: The test surface was machined with a new crosscut tungsten carbide bur in a laboratory hand piece at 30,000 rpm.

Sandblasted surface: The test surface was sandblasted at 5 bar air pressure using 60/80 grit at a constant distance of 5cm for 10 seconds.

Test specimen production

The metal primer is a functional monomer containing MEPS thiophosphoric methacrylate. The test surfaces of specimens treated with metal primer were fully wetted and allowed to dry before application of the composite resin. Once the metal primer had been applied the process for applying the composite resin was the same for all groups. The protocol and sequence of bonding the composite resin was dictated by the manufacturer's instructions. Initially each test surface had foundation opaque composite resin applied and polymerised in the specified light box for 1 minute. Subsequently opaque composite resin shade A1 was applied and polymerised for 1 minute in the light box. This was repeated to produce two layers of the opaque composite. Each dumbbell was then placed within a clear alignment jig and dentine composite resin placed between them and the dumbbells brought together. The alignment jig was such that when the dumbbells were brought together, a consistent 2 mm of composite resin was produced between them. A total of 84 test specimens were produced.

Statistical analysis of data

Data were extrapolated from the recordings and analysed using Stata version 9.2. Significance was pre-determined at p=0.05. The Shapiro-Wilk test was used for distribution analysis and ANOVA for variance analysis.

RESULTS

Tensile testing

71 specimens underwent testing, the mean tensile bond strength (Table 1) varied from 0.29 Mpa to 24.93 Mpa and the standard deviation 0.25 to 4.1 Mpa. Groups 1 (unprimed beaded surface) and 4 (primed machined surface) had one specimen per group fail before test data could be retrieved due to incorrect handling of the test equipment. 11 of the test specimens in group 3 (unprimed machined surface) failed during storage or in handling, and data was unable to be collected.

Figure 1 shows the greatest mean tensile bond strength was produced by group 6 (primed sandblasted surface) at 24.93Mpa whilst the weakest mean tensile bond strength was produced by group 3 (unprimed machined surface) at 0.29MPa. The peak tensile bond strength achieved was 32.36 Mpa by group 4 (primed machine surface) and this group also had the largest variation in the data obtained varying from 10.6 to 32.36 MPa. On the basis of the Shapiro-Wilk test all data was normally distributed, however data from group 3 (unprimed machined surface) was omitted as such a small sample size (Table 1) was shown to skew the data analysis.

Initial analysis of the data from the groups (Table 2) confirmed that there were statistically significant differences between the groups (F= 54, p= 0.0001). Group 1, the unprimed sandblasted specimens and group 5, the unprimed beaded specimens were analysed for significant differences and it was found that there was no statistically significant difference between these groups (F=0.001, p=0.98).

Analysis of the three groups treated with Metal Primer II, beaded, machined and sandblasted allowed for between

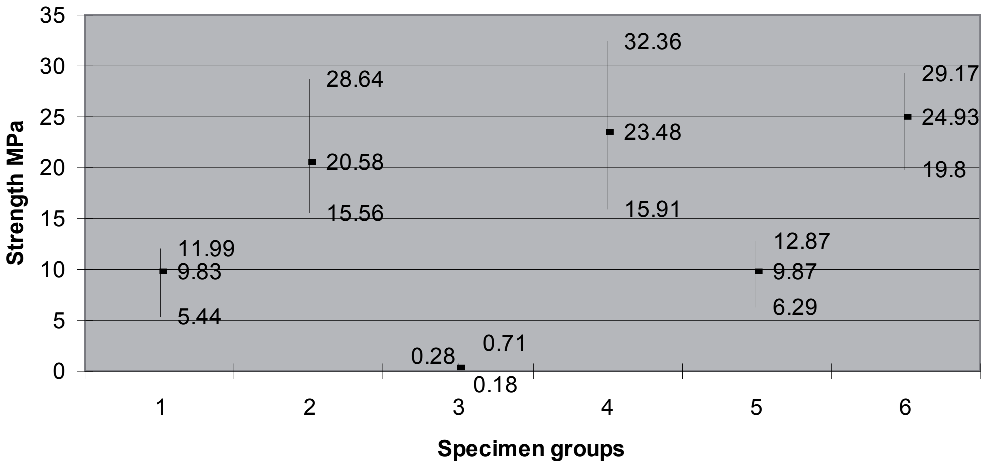
Table 1. *Summary statistics of experimental data.*

Group	*n*	*Mean (N)*	*SD*	*Mean Bond strength (MPa)*
1	11.00	278.27	51.88	9.83
2	12.00	582.08	116.06	20.58
3	4.00	8.25	7.89	0.28
4	14.00	663.57	161.75	23.48
5	15.00	279.20	55.35	9.87
6	15.00	705.00	91.50	24.93

Group 1 = Beaded test surface
Group 2 = Beaded test surface with Metal Primer II
Group 3 = Machined test surface
Group 4 = Machined surface with Metal Primer II
Group 5 = Sandblasted test surface
Group 6 = Sandblasted test surface with Metal Primer II

Table 2. *Analysis of Variance*

Source	*SS*	*df*	*MS*	*F*	*Prob> F*
Between groups	2352532.83	4	588133.209	54.00	0.0000
Within groups	675304.927	62	10892.015		
Total	3027837.76	66	45876.3297		



1 Beaded, 2 Beaded with primer, 3 Machined, 4 Machined with primer, 5 Sandblasted, 6 Sandblasted with primer.

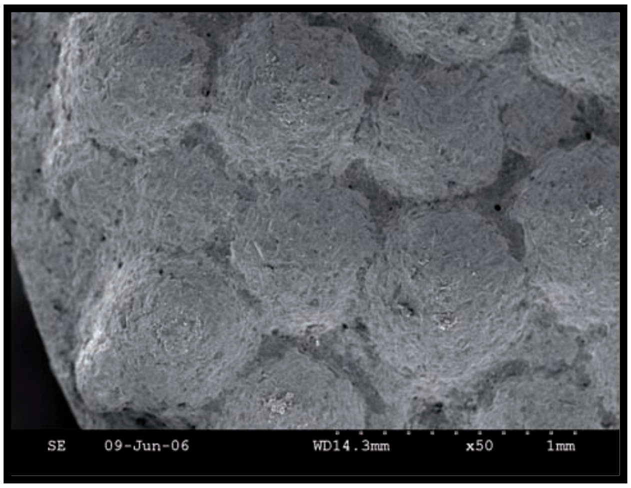
Figure 1. *Dataset of range and mean strength MPa of specimen groups.*

group variance to be tested. One way ANOVA analysis of these results showed that there was no statistically significant difference between the beaded and machined groups (F=3.94, p=0.14) and the machined and sandblasted groups (F=1.14, p=0.64). There was however a statistically significant difference between the tensile bond strengths achieved between the beaded and sandblasted groups (F=9.25, p=0.01).

Collating the data from the groups that were not treated and those that were treated with Metal Primer II, allowed analysis of the effect of the application of the primer. This confirmed that there was a statistically significant difference between the tensile bond strengths of groups treated with Metal Primer II and the groups not treated with Metal Primer II (F=197.97, p=0.0001). Metal Primer II increased the strength achieved across all groups that had been treated.

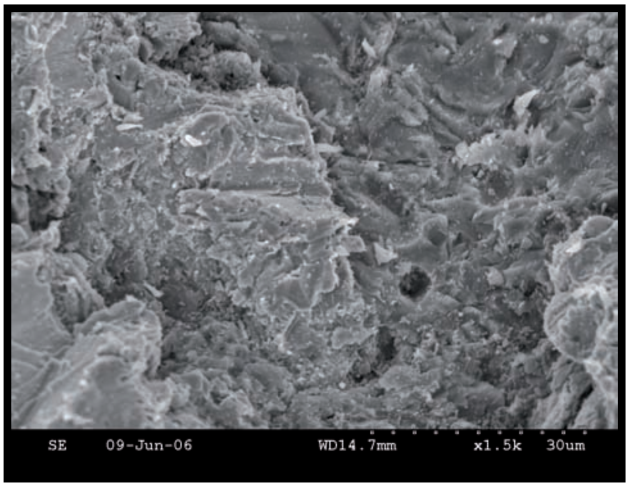
Scanning electron microscopy

Low magnification examination of the beaded surface showed very few interstitial spaces with no undercuts present (Fig. 2). At high magnification (Fig. 3) its surface mimicked that of the sandblasted surfaces both having a roughened appearance with micro undercuts and crevices evident. The machined surface (Fig. 4) contrasted this with a featureless, smooth appearance. With the application of



The surface shows a beaded pattern, however no spaces or undercut areas exist between the individual beads.

Figure 2. *SEM of beaded surface at x50*

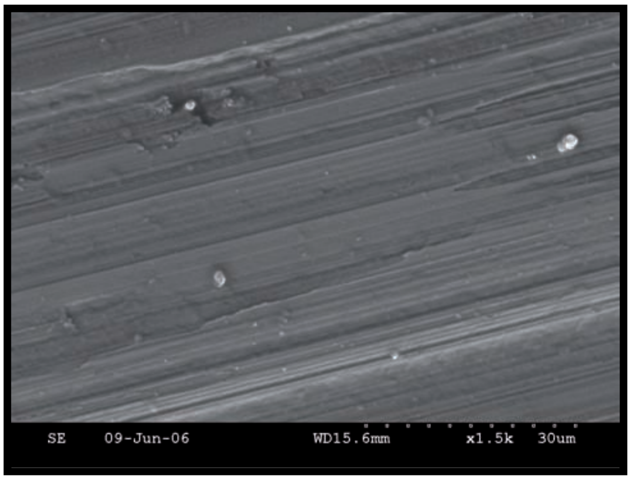


The sand blasted surface of the beaded specimen with micro undercuts and crevices present.

Figure 3. *SEM of beaded surface at x1500*

The machined surface is relatively featureless and smooth with no obvious mechanical undercuts. Striations present are interpreted as bur markings.

Figure 4. *SEM of machined surface at x1500*



The primed machined surface is relatively featureless. Brush strokes can be seen as the horizontal striations.

Figure 5. *SEM of machined/primer surface 1500x*

Metal Primer II a layer of resin over the smooth surface was detected with striated brush marks being seen (Fig. 5).

DISCUSSION

This study tested to failure in tension the bond strength of cobalt-chromium alloy specimens to composite resin. The unprimed machined group provided limited experimental data as several failed during storage; the four specimens that underwent testing provided the lowest bond strengths of 0.25 MPa (Table 1). This contrasts with other studies where bonding between titanium and acrylic resin has shown comparable tensile bond strengths with a sandblasted and machined surface[13]. Within the remit of this study, however, an untreated sandblasted surface produced mean bond strength of 9.87 MPa. SEM investigation showed that the sandblasted surface appeared roughened with microscopic undercuts which could be engaged by bonding material and provide a degree of mechanical retention (Fig, 3). In contrast the machined surface appeared uniformly smooth (Fig. 4). This may explain the failure of most of the machined specimens, as there appeared to be only minimal mechanically retentive undercuts. Following this work, machining cobalt-chromium alloy with a tungsten carbide bur cannot be recommended as it provides a surface finish that provides no or only minimal mechanical retention and consequently no appreciable bond strength.

The beaded surface was expected to produce the greatest bond strength. However, the results did not verify this hypothesis and contradicted previous studies. Tarozzo *et al*[1] showed that 0.6mm retention beads to be the most retentive system for bonding composite resin to nickel-chromium alloy and produced bond strengths of 63 MPa, far in excess of the bond strengths achieved in this study. It was not clear from their study as to the method of placement of the retention beads, as importantly the number and pattern of beads has been shown to influence bond strength[1]. In contrast to this, Tulunoglu and Oktemer[16] found no difference in bond strengths produced with composite resin and nickel-chromium alloy with 4-META, silicoating and bead retention surfaces.

The unprimed sandblasted and beaded surfaces produced similar results with no statistically significant difference between the two, with mean bond strengths of 9.87 MPa and 9.83 MPa respectively. These surfaces appeared similar being a sandblasted surface with undercuts and crevices. The retention beads should have provided a larger surface area and together with macroscopic undercuts produced higher bond strengths but this was not the case due in part to the close packing of the beads (Fig. 2). The loss of surface detail may have been due to the coarse investment material and inter-bead spacing, as undercuts were not reproduced on the alloy specimen. Shue *et al*[15] showed that the largest tensile bond strengths were produced with the retention beads the diameter of the bead apart.

The effect of applying metal primer to all groups was to increase the bond strengths achieved. In all the metal primer treated groups, the failure was a combination of cohesive and adhesive failure compared to the untreated groups where without exception all failures were adhesive.

The increase in bond strength was most evident with the machined surface groups. Untreated, the specimens were unable to be tested. However, when treated with metal primer the mean bond strengths achieved were 23.48 MPa (unprimed 0.28 MPa). This improvement in bond strengths was such that the primed machined surface was not statistically significantly different from the most favourable group, the sandblasted surface (24.93 MPa). The beaded surface when treated with metal primer produced the weakest bond strengths (20.58 MPa). This is a reversal in order when compared to untreated groups and appears to show that the retention beads provide no advantage in bond strengths. Compared to the earlier published literature[3] the bond strengths obtained in this study are a considerable improvement. Other authors have published bond strengths in excess of those achieved in this study; however it should be noted these are shear bond strengths which can be expected to be higher than tensile bond strengths.

When comparing the scanning electron microscope photomicrographs of the three surfaces the addition of metal primer has no effect on the surface topography. Untreated the machined surface (Fig. 4) is a flattened surface and with the addition of the metal primer (Fig. 5), it appears as a painted surface with brush marks clearly evident. When viewing the sandblasted surfaces, metal primer appears not to be evident, although it is postulated that the resin flows into the interstitial spaces. A change in surface topography is therefore not attributed to the increase in bond strengths achieved by the use of metal primer.

The greatest bond strengths within the limits of this study were produced by the machined and sandblasted surface treated with metal primer with no statistically significant difference between them. However, the distribution of these bond strengths are such that even though there is no statistically significant difference, the greater scatter of bond strengths leads to the conclusion that the machined surface produces a less predictable bond strength. The grouping of the sandblasted specimens was more closely clustered suggesting a more reliable bond strength which is clinically significant.

CONCLUSIONS

Within the limits of the study it has been shown:

The surface preparation of the cobalt-chromium alloy did influence tensile bond strengths with composite resin.

Metal Primer II increased tensile bond strengths for all groups tested.

The sandblasted surface treated with Metal Primer II is recommended for the bonding of composite resin to cobalt chromium alloy.

ACKNOWLEDGEMENTS

Thanks are due to Mr Richard Mallet, Mr Alan Black, Mr Ken Brady and finally Dr Martyn Sherriff for their assistance.

This study was awarded the Society for the Study of Prosthetic Dentistry Research Award 2006 and was presented at the Society for the Study of Prosthetic Dentistry Research 54th annual conference in Newcastle-Upon-Tyne 2007 and awarded the Schottlander prize 2007.

MANUFACTURERS DETAILS

- Instrom 1193 Universal testing machine, Instrom Ltd, Wycombe, UK.
- Cast acrylic rods, RS Products Ltd, Northants, UK.
- Croform Excel alloy, Davis Schottlander and Davis Ltd, Letchworth, UK.
- Veneer-Loc 0.5mm beads, George Taub Products, New York, USA.
- Crosscut tungsten carbide, bur no 500 104 274 Bracon Ltd, Etchingham, UK.
- Metal Primer II GC Corp, Tokyo, Japan.
- Gradia composite resin, GC Corp, Tokyo, Japan.
- GC Labolight III, GC Corp, Tokyo, Japan.

ADDRESS FOR CORRESPONDENCE

Mr David Newsum, Specialist registrar in Restorative Dentistry, Orthodontics and Restorative Dentistry, Glenfield Hospital NHS Trust, Groby Road, Leicester, LE3 9QP, United Kingdom. E-Mail: d_newsum@hotmail.com

REFERENCES

1. Tanaka, T., Nagata, K., Takeyama, M., Atsuta, M., Nakabayashi, N., Masuhara, E. 4-META opaque resin: a new resin strongly adhesive to nickel-chromium alloy. *J. Dent.Res.* 1981; **60**:1697-1706.
2. Tanaka, ., Fujiyama, E., Shimizu, H., Takaki, A., Atsuta, M. Surface treatment of non-precious alloys for adhesion-fixed partial dentures. *J. Prosthet. Dent.* 1986; **55**: 456-462.
3. Barzilay, I., Myers, M.L., Cooper, L.B., Graser, G.N. Mechanical and chemical retention of laboratory cured composite to metal surfaces. *J. Prosthet. Dent.* 1988; **59**:131-137.
4. Matsumura, H., Kawahara, M., Tanaka, T., Atsuta, M. Surface preparations for metal frameworks of composite resin veneered prostheses made with an adhesive opaque resin. *J. Prosthet. Dent.* 1991; **66:**10-15.
5. Yoshida, K., Taira, Y., Matsumura, H., Atsuta, M. Effect of adhesive metal primers on bonding a prosthetic composite resin to metals. *J.Prosthet Dent.* 1993; **69**:357-362.
6. Yoshida, K., Sawase, T., Watanabe, I., Atsuta, M. Shear bond strengths of four resin cements to cobalt chromium alloy. *Am.J.Dent.* 1995; **8**:285-288.
7. Kern, M., Thompson, V.P. Durability of resin bonds to a cobalt-chromium alloy. *J.Dent.* 1995; **23**:47-54.
8. Yoshida, K., Kamada, K., Tanagaw, M., Atsuta, M. Shear bond strengths of three resin cements used with three adhesive primers for metal. *J. Prosthet.Dent.* 1996; **75**:254-261.
9. Orchard, N..A, Howlet,t J.A., Davies, E.H., Pearson, G.J. Adhesive composite resins for artificial teeth: a laboratory investigation of bond strength to a cobalt-chromium alloy. *Biomater.* 1997; **18**:935-938.
10. Behr, M., Rosentritt, M., Gröger, G., Handel, G. Adhesive bond of veneering composites on various metal surfaces using silicoating, titanium-coating or functional monomers. *J.Dent.* 2003; **31**:33-42.
11. Mudford, L., Curtis, R.V., Walter, J.D. An investigation of debonding between heat-cured PMMA and titanium alloy (Ti-6AL-4V). *J.Dent.* 1997; **25**:415-421.
12. Salonga, J.P., Matsumura, H., Yasuda, K., Yamabe, Y. Bond strength of adhesive resin to three nickel-chromium alloys with varying chromium content. *J.Prosthet.Dent.* 1994; **72:**582-584.
13. Rhadi, A., Juszczyk, A.S., Curtis, R.V., Radford, D.R., Clark, R.K.F. Effect of GC Metal Primer II on bond strength of heat cured acrylic resin to titanium alloy (Ti-6Al-4V) with two different surface treatments. *Eur.J.Prosthet.Rest.Dent.* 2008; **16**: 132-137.
14. Tarozzo, A., Chiarello De Mattos, M.G., Riberi, .F, Semprini, M. Comparison of retentive systems for composites used as alternatives to porcelain in fixed partial dentures. *J.Prosthet.Dent.* 2003; **89**:572-578.
15. Shue. S-L., Nicholls, J.I., Townsend, J.D. The effect of metal retentive designs on resin veneer retention. *J.Prosthet.Dent.* 1987; **58**:297-305.
16. Tulunoglu, I.F., Oktemer, M. Tensile strength and micro leakage of the bond between a nickel-chromium alloy and a visible light-cured resin composite: effect of 4-META, silicoating and bead retention. *Quintessence Int.* 1997; **28**:447-451.