RESEARCH ARTICLE

THE EFFECTS OF SURFACE TREATMENTS AND BONDING AGENTS ON THE BONDING OF METAL-INDIRECT LABORATORY COMPOSITE MATERIALS

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ABSTRACT

Background: The aim of this in-vitro experimental study was to evaluate the shear bond strength between the base metal Co-Cr alloy and laboratory-cured composite with mechanic retention, chemical treatment and the combination of both treatments on a metal framework.

Materials and Methods: 200 alloy discs (Co-Cr) were prepared, half of the specimens were prepared with retention beads and the other half was grind. All test specimens were divided into 5 groups; control, sandblasted, sandblasted and Metal primer II (GC Co., Tokyo, Japan), sandblasted and Metal photo primer (Shofu, Kyoto, Japan), Rocatectribochemical silica coating system (3M ESPE, Seefeld, Germany) are used for each group. Then; Gradia opaque and Gradia indirect laboratory composites (GC Co., Tokyo, Japan) were applied to all groups. Half of the specimens are subjected to thermal cycling (TC) after the application of Gradia. Shear bond strength of each specimen was tested in an universal testing machine and fractured specimens were examined with SEM.

Results: While the lowest bond strength was obtained in with both control groups of retention beads with TC or without TC (1.37 MPa, 1.19 MPa), the highest bond strength was obtained in group that used Metal primer II with TC (21,45 MPa).

Conclusion: Indirect laboratory composites shows higher shear bond strengths when first sandblasted and then used metal primer for bonding.

INTRODUCTION

Indirect laboratory composite materials have been introduced as an alternative veneering material to porcelain as well as conventional acrylic resins. The advantages of veneering composite materials include easy handling, excellent aesthetics, hardness similar to that of the natural tooth enamel and ability to be readily repaired (Barzilay *et al.*, 1988). Moreover, the ability of these resins to be used as pontics for resin bonded fixed partial restorations, overlay material for removable partial prosthesis, veneered crowns and other types of fixed partial dentures can be included (Kolodney *et al.,* 1992)..However, the disadvantage is the relatively low bond strength of veneering composite to metal framework (Rose *et al.*, 1991; Mukai *et al.*, 1995). Several methods have been explored in an attempt to maximize the bonding of resin composite to metal. These are; can be classified as macro, micromechanical, chemical, or a combination of these (Cobb *et al.*, 2000).

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Traditionally, the bonding between laboratory-polymerized resin composite and metal is based on macromechanical retention (Leinfelder *et al.,* 1994). However this process produced a bulkier framework that was difficult to opaque because of pooling around the beads, which results in decrease of retention. Electrolytic etching technique developed by Livaditis *et al*., 1982; and this technique is sensitive and only some nickel-chromium and cobalt- chromium alloys can be successfully etched (Laufer*et al.,* 1988). Sandblasting with alumina powder has also been used to clean the alloy surfaces and to achieve both micro retentive topography and increased surface area. It results in a highly activated surface, which can be shown by the increased wettability of this material (Kern *et al.,* 1994; Tanaka *et al.,* 1986). Various adhesive metal primers have also been studied to enhance the bond strength of the composite resins to different metal surfaces (Yoshida *et al*., 1997; Yoshida *et al*., 1999). The main advantage of these adhesive agents is the simplicity of their application on the sandblasted alloy surface without the need for any other specific media (Seimenis*et al.,* 2006). The phosphoric and carboxylic acid functional monomer also used in most of these adhesive opaque resins and promoters was effective in bonding composite resin in base dental alloys (Almilhatti *et al.,* 2003;

Watanabe*et al.*, 1999). The aim of this study was to analyses the shear bond strength between the base metal Co-Cr alloy and laboratory-cured composite with mechanic retention, chemical treatment and the combination of both treatments on a metal framework. The effect of thermal cycles on the durability of the bond was also be investigated.

MATERIALS AND METHODS

The shear bond strength test procedures were performed according to ISO 10477. The materials used in this study are summarized in Table 1. Two-hundred autopolymerising acrylic resin (GC Pattern Resin LS, GC Europe) rivet-shaped patterns were formed in an addition silicone (Elite Double, Zhermack, Italy) mould with the following dimensions: disk 3 mm thick and 7 mm in diameter, pin 6 mm long and 3 mm in diameter. Then retention beads (GC Retention Beads II SSS, GC Europe) were sprayed onto the adhesive layer. All acrylic resin specimens were invested under vacuum with phosphate-bonded investment (Bellavest SH, Bego, Germany). After burnout at the temperatures specified by manufacturer, the specimens were centrifugally casted in Co-Cr alloy (Wironit, Bego, Germany) according to the manufacturer's instructions.

The remaining parts of the investment were removed by sandblasting (Basic Classic; Renfert GmbH, Hilzingen, Germany) with 110 μ malu minium oxide (Korox 110, Bego, Bremen, Germany). The alloy patterns were randomly divided into two major groups; with retention beads (G1) and without retention beads (G2). All the metal disks without retention beads were first polished with No. 600 SiC abrasive paper to create a flat surface and then ultrasonically cleaned for 10 minute in a distilled water bath (Whaledent Biosonic, NY, USA) and air dried and randomly divided into five subgroups of 20 specimens. G1C and G2C were control group, the metal specimens were left untreated. For G1S and G2S were the sandblasted group; the metal specimens were airborne- particle abraded using 110 μm Al_2O_3 particles (Bego, Bremen, Germany). Airborne-particle abrasion was performed for 10 seconds at 2.5 Atm air pressure, at a 10-mm nozzle metal surface distance. For G1P and G2P group, after sandblasting the metal surface was treated with primer (Metal primer II, GC Corporation, Japan) with a single brush application, which was allowed to dry for 1 min. For G1PP and G2PP, after sandblasting the metal surface was treated with primer (Metal photo primer, Shofu, Japan) with a single brush application, which was allowed to dry for 1 min. For G1R and G2R; Airborne-particle abrasion was performed initially with 110 μm Al_2O_3 particles (Rocatec Pre; 3M ESPE, Seefeld, Germany) for 10 seconds to clean the surface. A second airborne-particle abrasion treatment was performed for 13 seconds using a special alumina powder (110 μm) with added silica particles (Rocatec Plus; 3M ESPE, Seefeld, Germany) to form a silica surface layer. Airborne-particle abrasion was performed at 2.8 Atm air pressure, at a distance of 10 cm. And then, silane-coupling agent (3M ESPE Sil; 3M ESPE, Seefeld, Germany) was applied and allowed to dry for 5 min. After surface preparation, polymerization procedures were performed according to the manufacturer's instructions. A thin layer of Gradiaopaquer (GC Co., Tokyo, Japan) was applied and light-cured for 10s each with GC Steplight SL-1 (GC Co., Tokyo, Japan). The veneer composite Gradia (GC Co., Tokyo, Japan) was layered in three steps with a total thickness of 3 mm, and every layer was light-cured for 10s with GC Steplight SL-1 (GC Co., Tokyo, Japan) and post-curing was performed

for 3 min with GC Labolight LV-III (GC Co., Tokyo, Japan). Half of the specimens were performed thermal cycling (TC) between 5°C and 55°C (±2) for 5000 cycles with a dwell time of 30 seconds in each bath, according to ISO 10477. All specimens were then submitted to shear bond strength test (Autograph AG-IS 5K-N SHIMADZU, Kyoto, Japon) at a crosshead speed of 1 mm/min until failure. The shear bond strength (SBS) values were obtained in Newton (N) and converted into MPa. The shear bond strengths were calculated according to the formula: B = F / S, (B: shear bond strength (MPa), F: load at fracture (N), and S: bonded surfaces area (mm2). Fracture surfaces were evaluated with a scanning electron microscope (SEM) (Quanta 450FEG, USA). During evaluation of the study data, NCSS (Number Cruncher Statistical System) 2007 Statistical Software (Utah, USA) was used. As the parameters showed compatibility to normal distribution, the One-Way ANOVA was used in intergroup comparisons of parameters, while the Post Hoc Tukey HSD test was used in comparisons of sub-groups. The results were evaluated at the significance level of P<0,01.

RESULTS

Shear-bonding resistance test evaluation

While the lowest bond strength was obtained in G2 a/b (1.37 MPa, 1.19 MPa), the highest bond strength was obtained in G1P b (21,45 MPa). There is no statistically significant differences between the groups G1P, G1PP and G1R3 with no thermal cycle application (TC). (P<0,01). There are no statistically significant differences between the groups G2P and G2R, G2PP and G2R with no TC. On the other hand, G2P showed statistically higher values than G2PP (P<0,01). G2C and G1C with TC were significantly lower than all of the other groups with TC. On the other hand, G2P with TC showed statistically higher values than all of the other groups (P<0,01). Except G1R without TC, all the groups with retention beads showed significantly higher values than the without retention beads groups. Except G2R, all the groups with retention beads showed significantly higher values than the without retention beads groups.

Fracture interface evaluation

Fracture surfaces were evaluated with a scanning electron microscope (SEM) (Quanta 450FEG, USA). Fracture surfaces were evaluated under ×160 and ×300 magnification to determine the nature of the failure (cohesive, adhesive, or combination) and the interfaces involved and were scored using the modified adhesive remnant index (Table III). The results were evaluated statistically. As the parameters showed compatibility to normal distribution, the One-Way ANOVA was used in intergroup comparisons of parameters, while the Post Hoc Tukey HSD test was used in comparisons of sub-groups, and the Student t test in comparisons of pair groups The results were evaluated at the significance level of P<0,01. According to the evaluation to modified ARI, on the 119 specimen, remmant found less then %50 on the metal surfaces (Table 4).

Scanning Electron Microscope Evaluation

Fracture surfaces were evaluated with a scanning electron microscope (SEM) (Quanta 450FEG, USA).White arrows show the retention bead, orange arrows show the opaque layer of composite. SEM images were supporting the ARI index results (Fig 1).

Table 1. The materials used in this study

Material	Component	Manufacturer
Wironit Co-Cr metal	%64 Co, %28.65Cr, %5 Mo	Bego, Bremen, Germany
Gradia İndirect composite	bis-(methacryloyloxy)-propoxycarbonylaminohexane-triazine-trionemonomer, Aluminoborosilikat Silica %75	GC Corporation, Tokyo, Japan

Table 2. Mean Shear Bond Strength (MPa) and standard deviation of the all group

	(a)Thermal Cycling (-)		(b)Thermal Cycling (+)	
	G1	G2	G1	G2
Control (C)	8,18±2,15	1,37±1,2	5,11±1,85	1,19±1,13
Sandb. (S)	9,1±1,43	5,61±0,88	11,72±1,71	4,7±0,6
Metal primer (P)	18,13±2,88	15,1±1,11	21,45±3,01	17,25±1,45
Photo primer (PP)	16,16±2,29	12,14±2,78	17,91±2,49	13,44±1,44
Rocatec (R)	15,95±1,62	14,69±3,01	15,26±1,54	12,56±2,31
F	44,85	90,26	81,27	166,46
p	0,0001**	0,0001**	0,0001**	0,0001**

F:Oneway ANOVA Test and Post Hoc Tukey HSD test was used. **P<0,01

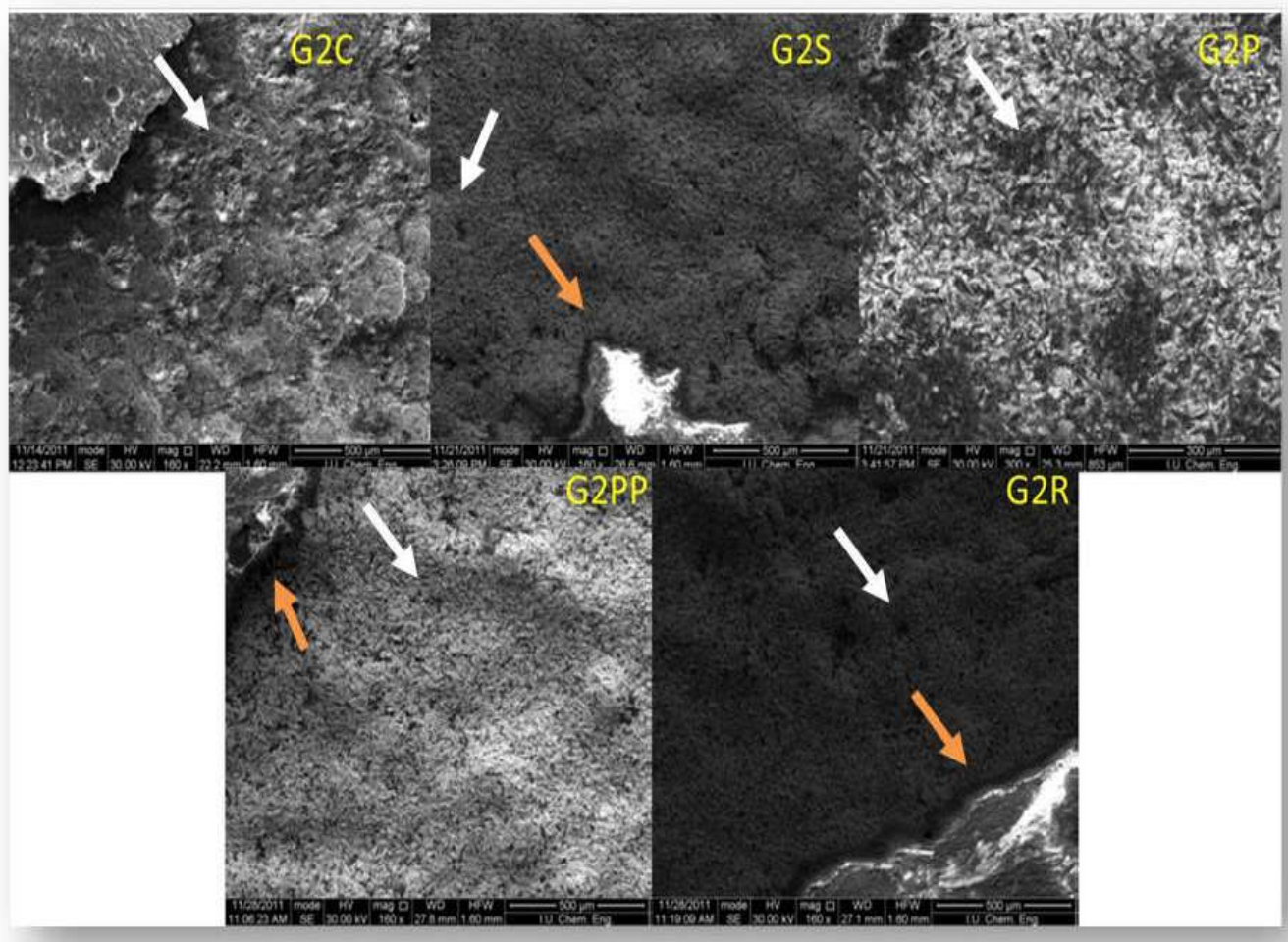**Fig. 1. Scanning electron microscope images with the magnification ×160. White arrows show the retention bead, orange arrows show the opaque layer of composite**

Table 3. Classification of the remnant index

Score	Remnant
0	No remnant on metal surface
1	Remnant on metal surface less than %50
2	Remnant on metal surface more than %50
3	All composite on metal surface

Table 4. Evaluation of composite remnant on metal surface according to Modified ARI

Groups	n	Score 0	Score	Score	Score
G1Ca	10	2	8	-	-
G1Cb	10	4	6	-	-
G1Sa	10	2	7	1	-
G1Sb	10	2	8	-	-
G1Pa	10	-	2	7	1
G1Pb	10	-	-	6	4
G1PPa	10	-	6	4	-
G1PPb	10	-	8	4	-
G1Ra	10	-	8	2	-
G1Rb	10	-	10	-	-
G2Ca	10	10	-	-	-
G2Cb	10	10	-	-	-
G2Sa	10	10	-	-	-
G2Sb	10	10	-	-	-
G2Pa	10	-	10	-	-
G2Pb	10	-	9	1	-
G2PPa	10	-	9	1	-
G2PPb	10	-	10	-	-
G2Ra	10	-	10	-	-
G2Rb	10	-	10	-	-
Total	200	50	119	26	5

DISCUSSION

The metal-composite bond strengths measured by shear test were evaluated in present study according to the minimum value of 5,5 MPa specified in ISO 10477 Standard. Excluding the group (G1C/G2C) that was subjected to sandpaper application, the groups that were subjected to sandpaper, sandblasting and thermal cycling, in all other groups (G1P/G2P; G1PP/G2PP; G1R/G2R; with or without TC the bond strength was above the value of 5 MPa. Matsumura *et al*., 2001; argues that the shear bond strength for clinical use of metal-composite restorations must exceed 10MPa value. The bond strength in all groups on which metal primer and tribochemical silica coating were applied was above the values specified in ISO 10477.

In present study, it was found that the joint use of sandblasting-primer and tribochemical silica coating application on metal surface increased the metal-composite bond more than individual use of the sandblasting application. Sandblasting with aluminium oxide increases the amount of aluminium and oxygen on the metal surface and reduces the initial amount of elements (Ohkubo *et al*., 2000). According to Kern *et al*., 1994; aluminium particles used in sandblasting reveal metal oxides on metal surface and thus provide formation of a passive film layer bonded to functional monomers of metal primers, and so increase the metal-composite bond strength. In present study, after thermal cycling, a reduction in the bond strength was observed on the samples, which were only subjected to the sandblasting surface treatment. Also, in the study carried out by Yanagida *et al.,* 2000; is stated that in the metal-composite bond strength, thermal cycling leads to a reduction in bond on the surface subjected to sandblasting and that a chemical bond must be provided in order to increase the bond strength after sandblasting. In all groups on which thermal cycling was not applied; G1Ca/G2Ca, G1Sa/G2Sa, G1Pa/G2Pa, G1PPa/G2PPa, the efficiency of the use of

retention beads increased the metal-composite bond strength. Although only in the group with retention beads within the group G1Ra/G2Ra an increase in number was observed in bond-strength, no significant difference was observed in statistical terms. In this case, we could say that use of retention beads increases the metal-composite bond strength independently from other surface treatments. There is an opinion that a micro leakage will occur in the interface between the mechanical retention otained by the individual use of retention beads and metal-composite resin in time (Kourtis, 1997). The chemical bond to the metal surface of the opaque layer limits the micro leakage occurred between polymerizing shrinkage and metal-composite due to nonconformity of thermal expansion (Imbery *et al.,* 1993; Tanoue *et al.,* 1998). The chemical bond requires a precise technique and any possible contamination, which may occur in the interface, reduces the metal-composite bond (Yoshida *et al.,* 2001). In present study, it was found out that the application of metal primer and tribochemical silica coating on metal surface with retention beads resulted an increase in the metal-composite bond strength (G1P,G1PP and G1R with TC). The obtained bond strength value was considerably above the bond strengths given in ISO 10477 and suggested by Matsumura for clinical use of metal-composite restorations (Matsumura *et al.,* 2001). Similar to the results of current study, the previous studies (Matsumura *et al.,* 2001; Taira *et al.,* 1995; Yoshida *et al.,* 2001). are also of the opinion that metal primer creates a bond with oxide layer on metal alloy surface and thus increases the metal-composite bond strength. In the study in which 3 different indirect laboratory composites (Solidex, Artglass, Signum) as metal photo primer and veneer material were applied on Ni-Cr alloy surface with retention beads in 150μ diameter, and the samples were subjected to 5000 thermal cycling, respectively bond strength values of 12.61MPa, 14.9MPa and 16.55MPa were obtained (Seimenis*et al.,* 2006). These results are lower than those values obtained in group G1PP with TC in present study (17.94MPa). This difference might be caused by the use of different alloy and indirect composite material in present study.

Although higher bond strength was obtained in the group in which Metal primer II (G1Pa) was applied among those groups in which metal primer and tribochemical silica coating with retention beads were applied, but not subjected to thermal cycling (G1P,G1PP,G1R); no significant difference between them was found. The bond strength obtained in these groups are considerably above those bond strengths given in ISO 10477 and suggested by Matsumura *et al*., 2001 for clinical use of metal-composite restorations. The highest bond strength in groups not subjected to thermal cycling was obtained in the group subjected to Metal primer II (G1Pa) application. No statistically significant difference was observed between the Group G2PP and G2R. Yoon *et al.,* 2007; applied 2 different composite resins (Sinfony and Gradia) on sandpapered Cp-Ti/Co-Cr alloy surface and examined the shear bond strength. In the study, 2 different primers were used as Metal primer II and Rocatec. 17.70MPa (Metal primer II) and 16.61 MPa (Rocatec) bond strengths were obtained. These values are similar to the values of the groups G2Pa and G2R5a in present study (15.1MPa, 14.69MPa). Seimenis *et al.,* 2006; discovered in their study that 1000 and 5000 thermal cycling has no effect on metal-composite bond strength. In present study it was found that thermal cycling treatment has no effect on metal-composite bond strength in groups on which Metal Photo Primer (G1PPb/G2PPb) was used and tribochemical

silica coating (G1Ra/G2Rb) was applied. This situation may prove that a stable metal-composite bond, which withstands humidity and temperature changes, is formed (Kim *et al.*, 2003; Luthy *et al.*, 1990) However, thermal cycling treatment increased the metal-composite bond strength in groups G1Pb/G2Pb in which Metal Primer II was used, independently from the use of retention beads. Double bonds of MEPS in Metal Primer II become polymerized with double bonds of UDMA in the interface and form a single copolymer structure. It is considered that thermal cycling in limited numbers restricts its own effect and even in some cases, increases the bond strength a little, and this increase may be related to relief of polymerization stresses of composite material (Kern *et al.*, 1994; Schneider *et al.,* 1997). Also, there is an opinion that the increase in metal-composite bond strength after thermal cycling may be related to improvement in the monomer variation degree of polymer material due to temperature rise (Knobloch *et al.,* 1999; Moulin *et al.,* 1999; Tanoue *et al.,* 2000).

After polymerization, polymerization of some monomers could not be completed. The missing polymerization may be completed with heat during thermal cycling and it may lead to an increase in the bond strength. Yoon *et al.,* 2007; stated that; although Metal primer II showed higher bond strength than Rocatec, differently from in present study, no statistically significant difference was observed between them. Similarly to current study, the highest bond strength among Metal primer II, Metal photo primer and silica coating treatments applied on metal surface obtained with Metal primer II (32.3MPa) in the study conducted by Kurtz *et al.,* 2005; and no significant difference was found between Metal photo primer (24.2MPa) and silica coating (26.2MPa). Higher bond strengths were derived from in present study, and it is thought that it is due to the different alloy selection and the change in the sample size. In present study as a result of examination of metal and composite bond surfaces after shear bond strength test, it was observed that the most frequently encountered type of failure is mixed-type failure where both adhesive and cohesive cracks are seen together.

In the SEM examinations of metal and composite surfaces after shear bond strength test, on samples with retention (G1Sa, G2Sb), the opaque deposits were observed between retention beads, their tips had a clean surface. And on sandblasted samples, a completely clean metal surface was observed. All these findings supports that use of retention beads increases the metal composite bond strength, independently from the use of primer. And in groups G1Pa and G2Pb in which the highest bond strength was reached, composite and opaque deposits were observed on the tips of the retention beads. While appearance of a typical sandblasted metal surface was perceived on sandblasted metal surfaces, also resin layers separated from composite through adhesive and cohesive cracks on the surface were seen. In the areas where there are cohesive cracks in resin, the fillers inside the composite structure were spotted clearly. Differences between the effects of the use of Metal Photo Primer, Rocatec and Metal Primer II on metal-composite bond strength may be related to difference of some physical properties like their chemical contents, physical structures, viscosity and wettability. In addition, it could be said that affinities of these three different surface treatments on sandblasted Co-Cr metal alloy surface and bonding performances are different.

Conclusion

Use of metal primer agents is a low-priced, option with a simple and short application procedure which does not require any special apparatus or technical precision, and it is successful in the increase of metal-composite bonding and which is appropriate for clinical use. As a result of in this present study study, it is recommended to use techniques, which combine the chemical and mechanical retention for a reliable bond between metal and composite.

Conflict of interest: The authors declare that they have no conflict of interest.

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