

Adhesive Bonding of Resin Composite to Various Ni-Cr Alloy Surfaces Using Different Metal Conditioners and a Surface Modification System

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Abstract

Purpose: This study evaluated the effect of three metal conditioners [Metal Photo Primer[®] (MPP), Cesead II Opaque Primer[®] (OP), Targis Link[®] (TL)], and one surface modification system [Siloc[®] (S)] on the shear bond strength (SBS) of a prosthetic composite material to Ni-Cr alloy.

Materials and Methods: Rivet-shaped specimens were cast, and three surface treatments were evaluated: Polishing (P); sandblasting with either 50 μ m (50SB) or 250 μ m (250SB) Al₂O₃. All products were applied to half of the specimens, while the other half remained without the materials. Veneering resin composite (8-mm diameter, 2-mm thick) was applied and light-exposed for 90 seconds in a laboratory light-curing unit. The specimens were stored in water at 37°C for 24 hours, and half were subjected to 500 thermal cycles consisting of water baths at 4°C and 60°C. All specimens were submitted to SBS test (0.5 mm/min) until failure. Failure patterns were determined using optical and scanning electron microscope (SEM) analysis. Data were analyzed by ANOVA and post hoc Tukey's test (preset alpha of 5%).

Results: The SBS values of OP and TL groups were higher than those of MPP and S within the 50SB treatment (p < 0.05). No significant difference in SBS was noted between OP and TL as well as between MPP and S. On the other hand, no significant differences were found among conditioners within the 250SB group (p > 0.05). The SBS values of MPP, OP, and S from the 250SB group were higher than those from 50SB (p < 0.05). No significant difference in SBS was noted among most groups with conditioners after thermocycling. The only exception was observed for MPP, which showed an increase in SBS after thermocycling (p < 0.05). Differences in SBS were noted among the groups with conditioners (p < 0.05), and no significant difference in SBS was noted between TL and OP groups, which showed the highest values among all within the P group. No significant difference was noted between MPP and S. Debonded surfaces showed adhesive failures predominantly located between metal surface and opaque resin.

Conclusions: The OP and TL conditioners and surface sandblasting with 250 μ m Al₂O₃ promoted the highest SBS between resin and the Ni-Cr metal surface.

An increasing number of composite veneering systems have recently been introduced for manufacturing artificial crowns, as an alternative to porcelain fused to metal. Several methods have been explored in an attempt to maximize the bonding of resin composite to metal. Such methods can be classified as macro, micromechanical, chemical, or a combination of these.¹ Traditionally, the bonding between laboratory-polymerized resin composite and metal is based on macromechanical retention.² During the last decade, a major advance was made in the development of new metal-resin bonding techniques that resulted in chemical bonding between resin and base metals. Several chemical methods that enhance resin veneer bonding to cast alloys were developed. One of the most important improvements in resin veneer bonding techniques was the addition of

 Table 1
 Materials used

Material	Identification	Abbreviation	Manufacturer	Components
Casting alloy	Wiron 99	NiCr	Bego, Bremen, Germany	Ni 65%, Cr 22.5%, Mo 9.5%, Nb 1%, Si 1%, Fe 0.5%, Ce 0.5%, C max. 0.02%
Metal conditioners	Metal Photo Primer	MPP	Shofu, Kyoto, Japan	4-acryloyloxyethyl trimellitate
	Targis Link	TL	Ivoclar, Schaan, Liechtenstein	10-methacryloyloxydecyl dihydrogen phosphate
	Cesead II Opaque Primer	OP	Kuraray, Osaka, Japan	10-methacryloyloxydecyl dihydrogen phosphate
Metal surface modification system	Siloc Bonding System	S	Heraeus Kulzer, Wehrheim, Germany	Methacryloxypropl trimethoxysilane
Opaque resin	C & B Opaque	0	Heraeus Kulzer, Wehrheim, Germany	Opaque OA3
Composite veneering material	Artglass	А	Heraeus Kulzer, Wehrheim, Germany	Dentin A3

4-methacryloxyethyl trimellitate anhydride (4-META) to acrylic resin.³ In 1981, Tanaka et al⁴ reported that the 4-META opaque resin was able to eliminate the need for mechanical retention on metal castings.

The first bonding technique was based on the Silicoater system, introduced in 1984 as a "system of molecular bond" between resins and dental alloy surfaces.⁵ In that system, the adhesion mechanism consists of the heat fusion of a microscopic layer of glass beads to the metal, and the composite is bonded with a silane coupling agent, which is known as pyrolytic silanization.⁶⁻⁸ In tribochemical coating, the heat is eliminated by the use of air-particle abrasion under pressure,^{6,9} while in a spark-erosion bonding system, sandblasting is eliminated.¹⁰ Another technique is based on metal surface modification by coating the surface with a layer of titanium nitride (TiN).¹¹

Advanced methods include the so-called "adhesive" resins with chemically active monomers that bond directly to airborneparticle-abraded alloy surfaces.¹² The availability of resin systems capable of chemically bonding to cast dental alloys simplified the procedures of metal surface preparation and contributed to the increased use of cast metal resin-bonded restorations. The new adhesive systems only require the airborne-particle abrasion of the metal surface before bonding.¹² The bonding agents lead to some mechanism of chemical bonding with the different alloys, so the micromechanical retention provided by airborne-particle abrasion increases considerably.¹³ The phosphoric and carboxylic acid functional monomer used in most of these adhesive opaque resins and promoters was effective for bonding resin composite to base dental alloys. With the use of appropriate adhesive metal primers, it is not only possible to eliminate the need for surface preparations of the metal frameworks before applying the light-cured veneering resins, but also to reduce the need for retentive devices on the metal substructure.14

Recently, several adhesive primers containing different functional monomers for bonding base metals to resin have become commercially available; however, the effects of these adhesive primers for base metals on the bond strength of metals to resin were not determined. Therefore, the purpose of this study was to evaluate whether surface pretreatment of Ni-Cr dental casting alloy with airborne-particle abrasion associated with three metal conditioners or with one surface modification system improves the bond strength of a light-activated resin composite material to base metal.

Materials and methods

All information about the materials used in this study is presented in Table 1. One hundred ninety-six rivet-shaped specimens were cast with Ni-Cr alloy using phosphate-bonded investment (Micro-Fine 1700, Talladium, Valencia, CA) using electric plasma arc, in an inert gas-conditioned casting machine (Discovery, EDG, São Carlos, Brazil), as previously described.¹⁵

After casting, the disk surfaces were gradually polished with silicon-carbide papers under water up to 600 grit. The alloy specimens were then washed in isopropyl alcohol for 10 minutes with ultrasonic agitation, were divided into three groups, and were submitted to three surface treatments: (1) polishing with 600-grit silicone-carbide paper (P)—control; (2) sandblasting with either 50 μ m Al₂O₃ (50SB); or (3) 250 μ m Al₂O₃ (250SB). Airborne-particle abrasion was performed perpendicularly to the surface at a distance of 5 mm at 0.5 MPa for 15 seconds (Basic Classic, Renfert, Germany).^{16,17}

After surface preparation was completed, each group (56 specimens) was divided into four subgroups according to the metal conditioners and to the metal surface modification system applied to the disk surfaces. In addition, 14 specimens of 50 μ m SB and 14 specimens of 250 μ m SB were left untreated and received only opaque and dentin resins to create the control groups (OD).

The metal conditioners evaluated in this study were Metal Photo Primer (MPP), Targis Link (TL), and Cesead II Opaque Primer (OP); the metal surface modification system was Siloc Bonding System (S) (Table 1). Each single-bottle metal conditioner was applied directly to the alloy surface with a brush following the manufacturers' instructions. For the Siloc Bonding System, one coat of primer (Siloc Pre) was applied to the specimen surfaces with a single brush application. The primed specimens were allowed to dry for 2 minutes before they were placed in an activation chamber (Silicoater MD, Heraeus Kulzer, Wehrheim, Germany), as recommended by the manufacturer. At the end of the cycle, the specimens were removed from the oven and were left at room temperature for 4 minutes. Afterward, one coat of the bonding agent (Siloc Bond) was applied with a single brush and was allowed to dry for 5 minutes.

Two thin layers of an opaque resin (O) were applied to the metal surfaces, and each layer was light-exposed for 90 seconds in a laboratory light-curing unit (Dentacolor XS, Heraeus Kulzer). Veneering resin was applied to the specimen surface using a cylindrical stainless steel split mold with an internal hole (8 mm in diameter, 2 mm thick). Two 1-mm thick layers of dentin composite (A) were inserted into the stainless steel mold. The specimen was removed from the mold, and each layer was then polymerized for 90 seconds in the Dentacolor XS light-curing unit, resulting in a total light exposure of 180 seconds. The polymerization procedures were performed according to the manufacturer's instructions. The opaque and dentin resins were then applied to all groups.

Seven specimens from each experimental group (n = 7) were stored in water at 37°C for 24 hours (T0), while the other seven specimens were stored in water at 37°C for 24 hours followed by 500 thermal cycles (T1), which consisted of water baths at 4°C and 60°C with a dwell time of 15 seconds in each bath. It has been shown that the combination between water storage and thermal cycling reproduces the in vivo conditions more accurately.¹⁸⁻²¹ All specimens were then submitted to shear bond strength test (Model 810, Material Test System, MTS Corp., Eden Prairie, MN) at a crosshead speed of 0.5 mm/min until failure.^{16,22} The shear bond strength (SBS) values were obtained in kgf and converted into MPa.

Fractured surfaces were evaluated with an optical stereomicroscope (Carl Zeiss, Jena, Germany) at 40×magnification to determine the failure pattern (cohesive, adhesive, or combination). In addition, a representative specimen surface of each experimental group was evaluated with a SEM operating at 20 kV (JSM-T33AO, JEOL, Tokyo, Japan). All metal surface treatments were also examined by X-ray energy-dispersive spectroscopy (EDS). The EDS analyses were performed with SEM (JSM-T33AO, JEOL) at 20 kV and live time of 100 seconds. The metal disk surfaces were examined before and after airborne-particle abrasion.

The SBS means and standard deviations (SD) were submitted to 3-way ANOVA (surface treatments, conditioners, and thermal cycling) at 95% confidence level. One-way ANOVA was performed to compare differences in bond strength between the conditioners and the metal surfaces from the control group—polishing (P). Levene's and Shapiro-Wilk's tests were performed to detect the homogeneity and normality, respectively (p > 0.05). Differences among means were determined by post hoc Tukey's test at a preset alpha of 5%. The OD group was not included in the statistical tests to avoid the possibility of masking the effect of the conditioners.



Figure 1 Shear stress measurements in MPa.

Results

Figure 1 summarizes the shear bond strength of all experimental groups. The shear bond strength values (MPa) and SDs of all groups at T0 and T1 are presented in Table 2. Forty-two thermocycled specimens without any treatment agent showed spontaneous adhesive failures before the mechanical testing and were discarded from the study. Levene's and Shapiro-Wilk's tests found p = 0.246 and p = 0.838, respectively, as probability values for the 3-way ANOVA. Regarding 1-way analysis, Levene's test of variance homogeneity detected p =0.932, while Shapiro-Wilk's test of error normality detected p = 0.097. Therefore, it was assumed that the ANOVA was not affected by the difference in variability among groups in both situations (p > 0.05). A double statistical interaction was observed between treatment and conditioner factors as well as between thermal cycling and conditioner (p < 0.05) (Table 3). The post hoc Tukey's test (Table 4) indicated that the shear bond strength values of OP (7.5 MPa) and TL (7.7 MPa) were higher than those of MPP (6.4 MPa) and S (6.0 MPa) within the 50 SB treatment (p < 0.05). No significant difference was noted

Table 2 Shear bond strength (MPa)

			Surface treatments								
	Thermal	I	Р		50 SB		250 SB				
Conditioners	cycle	Mean	SD	Mean	SD	Mean	SD				
OD	TO	_	_	3.4	0.36	5.1	0.96				
	T1	-	-	1.9	0.46	6.2	0.98				
MPP	TO	1.3	0.64	5.1	0.79	7.6	0.68				
	T1	-	-	7.7	1.44	7.9	0.82				
OP	TO	2.3	0.57	6.7	1.07	8.6	1.33				
	T1	-	-	8.4	1.21	9.2	0.78				
S	TO	0.7	0.54	5.7	0.79	8.3	0.99				
	T1	-	-	6.2	1.17	9.7	1.17				
TL	TO	2.6	0.55	7.9	1.04	8.7	1.52				
	T1	-	-	7.6	1.69	8.5	1.44				

SD, Standard deviation.

Table 3 Analysis of variance (ANOVA) results

Source of variation	df	Mean squares	F	p	
Three- way ANOVA					
Treatment (A)	1	76.03	56.67	< 0.001*	
Thermocycling (B)	1	19.36	14.43	<0.001*	
Conditioner (C)	3	8.91	6.64	<0.001*	
(A) × (B)	1	2.42	1.80	0.183	
$(A) \times (C)$	3	5.95	4.43	0.006*	
(B) × (C)	3	4.31	3.21	0.026*	
(A) \times (B) \times (C)	3	3.54	2.64	0.054	
Residual	96	1.34	-	-	
One-way ANOVA					
Conditioner	3	5.67	16.99	<0.001*	
Residual	24	0.33			

Values followed by asterisk are statistically significant (p < 0.05).

between OP and TL as well as between MPP and S. On the other hand, no significant differences were found among conditioners within the 250 SB group (p > 0.05). The mean values of MPP, OP, and S from 250 SB group were higher than those from 50 SB (p < 0.05) (Table 4). The post hoc Tukey's test demonstrated no significant difference in shear bond strength values among groups with conditioners regarding the interaction between thermal cycling and conditioners. The only exception was observed for MPP, which showed an increase in flexural bond strength between T0 and T1 (from 6.3 MPa to 7.8 MPa) (p < 0.05) (Table 5). One-way ANOVA indicated significant differences in shear bond strength among groups with conditioners (p < 0.05) (Table 3) and no significant difference in shear bond strength values was noted between TL (2.6 MPa) and OP (2.3 MPa) groups, which showed the highest values among all for treatment P, based on Tukey's post hoc test. No significant difference was noted between MPP (1.3 MPa) and S (0.7 MPa) (Table 6). Debonded surfaces showed adhesive failures predominantly located between metal surface and opaque resin. The SEM analysis of metal surfaces revealed differences in metal surface morphology (Figs 2-4), and the EDS analysis detected the presence of aluminum oxide particles on the airborne-particle-abraded metal surface.

Discussion

The bond strength between metal and resin composite can be achieved by using several methods capable of creating macro, micromechanical, or chemical retentions or a combination of these.¹ In this study, two processes of resin/metal bonding were evaluated: systems containing active monomers (MPP, OP, and TL), which consist of molecules characterized by the presence of radicals with specific properties, so they are able to bond chemically to the oxide layer on the surfaces of the basic alloys, and a system defined as pyrolytic silanization (S).

The metal/resin shear bond strength values demonstrated by both control groups 50 SB/OD and 250 SB/OD, which rely solely on mechanical retention of the opaque resin to the metal surface, showed a trend toward lower values than the values observed when the same treated surfaces received the metal conditioners (Fig 1). Such a trend suggests that the contribution of the mechanical retention created by the airborne-particle abrasion with aluminum oxide (mainly in group 250 SB and excepting group 50 SB/T1) to the bond strength to metal exceeded the chemical bond of the conditioners to the metal ions of the Ni-Cr alloy of experimental groups polished with 600grit SiC papers. The chemical bond showed remarkable results for conditioners OP and TL, which promoted shear stress values of 2.3 MPa and 2.6 MPa, respectively. According to Behr et al,²³ the resin/metal interface of a typical restoration in the anterior region should at least bear a tension of around 10 MPa after thermal cycling or longer periods of storage in water, regardless of the metal used. In this study, the chemical bond was apparently more susceptible to hydrothermal stress than the mechanical retention, evidenced by complete displacement of the opaque resin from the metal after thermal cycling in Group P/T1 (Table 2). Therefore, the mechanical and chemical bonding mechanisms are crucial as they create a stable bond and improve the bond strength and durability of restorations.

The anchoring effect of mechanical bonding is related to metal surface roughness and preparations applied to the metal surface.²⁴ The morphological changes observed on the metal surfaces after sandblasting may be attributed to the differences in the size of the aluminum oxide particles, as pressure, time, and distance of airborne particle abrasion from the metal surfaces were the same in all groups. The effects of

		Mean		50 SB				250 SB		
Treatment	Conditioner		MPP	OP	S	TL	MPP	OP	S	
50 SB	MPP	6.4								
	OP	7.5	0.152							
	S	6.0	0.987	0.014*						
	TL	7.7	0.045*	1.000	0.003*					
250 SB	MPP	7.7	0.046*	1.000	0.003*	1.000				
	OP	8.9	< 0.001*	0.049*	<0.001*	0.164	0.160			
	S	9.0	< 0.001*	0.030*	<0.001*	0.109	0.106	1.000		
	TL	8.6	< 0.001*	0.212	< 0.001*	0.487	0.481	0.999	0.993	

Table 4 Tukey analysis for the interaction between treatment and conditioner

Values followed by asterisk are statistically significant (p < 0.05).

			ТО				T1		
Thermocycling	Conditioner	Mean	MPP	OP	S	TL	MPP	OP	S
ТО	MPP	6.3							
	OP	7.6	0.074						
	S	7.0	0.776	0.857					
	TL	8.3	< 0.001*	0.717	0.057				
T1	MPP	7.8	0.023*	1.000	0.612	0.919			
	OP	8.8	<0.001*	0.124	0.002*	0.958	0.293		
	S	8.0	0.008*	0.994	0.374	0.988	1.000	0.516	
	TL	8.0	0.004*	0.978	0.273	0.997	0.999	0.638	1.000

Table 5 Tukey analysis for the interaction between thermocycling and conditioner

Values followed by asterisk are statistically significant (p < 0.05).

airborne-particle abrasion were clearly noted on the SEM analyses of 50 SB and 250 SB groups (Figs 2–4). Based on the results of the interaction between treatments and conditioners (Table 2), no significant difference in shear bond strength values was noted between OP and TL when the metal conditioners were applied to the surfaces of 50 SB group. Such conditioners also promoted higher shear bond strength values than MPP and S.

On the other hand, no significant difference in shear bond strength was noted among metal surfaces treated with metal conditioners in 250 SB groups, but the use of MPP, OP, and S in the 250 SB group resulted in higher shear bond strength values than the values promoted by the same conditioners in the 50 SB group. This suggests that the increase in mechanical retention due to the use of 250 SB compensated for the differences in bond strength values promoted by different metal conditioners on 50 SB metal surfaces. These findings agree with those of Mukai et al.²⁵ who showed that airborne-particle abrasion with Al₂O₃ particles improved the effectiveness of the metal surface area, surface energy, and resin/metal bond strength. Another important consideration about the effect of airborne-particle abrasion also directly related to the effectiveness of the conditioners is the capacity of the Al₂O₃ particles to oxidize the surface of the Ni-Cr alloy by forming a passive film composed of Ni and Cr oxides.¹⁶ Physical and chemical bonding mechanisms involve molecular interaction between the adhesive and the substrate, while mechanical bonding does not require this type of interaction at the interface.^{26,27}

The effectiveness of the conditioners OP and TL may also be attributed to the fact that phosphoric acid group has chemical affinity to the aluminum oxide particles arrested on the metal

 Table 6 Tukey analysis for the interaction between conditioners for polishing treatment

Conditioner	Mean	MPP	OP	S
MPP	1.3			
OP	2.3	0.018*		
S	0.7	0.227	< 0.001*	
TL	2.6	0.001*	0.670	<0.001*

Values followed by asterisk are statistically significant (p < 0.05).

surface as a consequence of airborne particle abrasion.^{27,28} Based on the EDS analysis, no presence of aluminum or oxygen was noted on the polished surface, while airborne-particleabraded metal surfaces clearly showed the presence of these particles. For Ohkubo et al,²⁹ airborne-particle abrasion with aluminum oxide substantially increased the quantity of aluminum and oxygen on the metal surface and diminished the initial quantity of original elements. However, the influence of aluminum on the bond mechanism is not well known. Cobb et al¹ observed changes in the surface morphology of gold allovs due to the different sizes of the particles and the pressure of the airborne-particle abrasion. They also found an increase in the amount of aluminum on the metal surface and its possible chemical affinity to functional monomers and phosphoric derivatives. For Kern and Thompson,³⁰ in addition to the microretention created, airborne-particle abrasion leaves aluminum particles on the metal surface that have an important role in the bonding mechanism created by bonding systems with functional monomers.

When analyzing the results of the interaction between thermal cycling and conditioners (Table 2), it was observed that thermal cycling had no significant effect when conditioners OP, TL, and S were used. The only exception was observed for the MPP group, which showed higher shear stress values after thermocycling. These results indicate that a restricted



Figure 2 SEM analysis for Ni-Cr alloy before airborne-particle abrasion.



Figure 3 SEM analysis for Ni-Cr alloy after airborne-particle abrasion with 50 μm Al_2O_3.

number of thermal cycles may even cause a slight increase in bond strength, probably because of the relief of polymerization stress or the increase in the degree of conversion of the composite resinous material caused by the high temperature from thermocycling.³¹ On the other hand, the use of conditioners resulted in no significant differences in shear bond strength values when specimens were submitted to thermal cycles (group T1), different from the results of other studies.^{16,23,29,32-34} On the other hand, the thermocycling effects in group 50 SB/OD/T1 were more significant than those in group 250 SB/OD/T1 when conditioners were not used (Fig 1).

The results of this study imply that the greater retention created by airborne-particle abrasion with $250 \ \mu m$ particles compensated more effectively for the effects caused by the hydrothermal stress. Thus, the number of cycles used in this study (500) may be considered effective, as also demonstrated by Crim and Garcia-Godoy,¹⁸ who compared different numbers of thermal cycles, and concluded that effects of thermal cycling were evident in 100 cycles. According to Lakatos et al³⁵ the effects of themocycling appeared after 100 or 200 cycles, although there is no general agreement about the significance of thermal cycling with regard to thermal ranges in the oral cavity.



Figure 4 SEM analysis for Ni-Cr alloy after airborne particle abrasion with 250 μm Al_2O_3.

The results of this in vitro study suggest that the use of chemical bonding systems combined with mechanical retention enhanced the bonding between a resin composite and Ni-Cr alloy. Such evidence implies that the clinicians might consider the use of techniques that combine chemical and mechanical retentions when reliable bonding is required between metal surface and resin composite. Thus, the clinician can be more confident when repairing composite veneering systems coupled to metals.

Conclusions

According to the results of this investigation, It was concluded that optimization of resin bond to Ni-Cr alloy may be achieved by associating the mechanical retention provided by airborne-particle abrasion with 250 μ m aluminum oxide particles with the chemical retention of the metal conditioners, mainly those that contain functional monomers derived from phosphoric acid (Cesead II Opaque Primer, and Targis Link).

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