

COMPARATIVE STUDY OF SHEAR BOND STRENGTH BETWEEN CP-TI / CO-CR ALLOY AND COMPOSITE RESINS

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Statement of problem. Composite resin-veneered metal restorations can be used as an alternative to porcelain-fused-metal restorations. But, because of the relatively low bond strength of veneering composite to metal framework, various surface treatment methods have been introduced to improve the bond strength.

Purpose. The object of this study was to compare the shear bond strength of different combinations of each of the two bonding systems and each of the two composite veneering resins to cp-Ti / Co-Cr alloy.

Material and methods. Two resin bonding systems (metal conditioner containing MEPS monomer, tribochemical silicoating system) and two composite resins (Gradia, Sinfony) were tested on cp-Ti and Co-Cr alloy. Then, according to manufacturers' instructions, resin bonding systems and composite resins were applied. All test specimens were divided into four groups for each alloy; I) sandblast + Metal Primer II + Gradia (MG), II) sandblast + Metal Primer II + Sinfony (MS), III) Rocatec + Gradia (RG), IV) Rocatec + Sinfony (RS). The shear bond strength was determined using a universal testing machine and all data were statistically analyzed with Mann-Whitney test and Kruskal-Wallis test at the significance level of 0.05.

Results. The mean (standard deviations) of shear bond strength according to the combinations of two bonding systems and two composite resins to cp-Ti arranged from 16.44 MPa to 17.07 MPa and the shear bond strength to Co-Cr alloy ranged from 16.26 MPa to 17.70 MPa. The result shows that the difference were not statistically significant.

Conclusion. The shear bond strengths of composite resins to both cast cp-Ti and Co-Cr alloy were not significantly different between the metal conditioner and the tribochemical silicoating system. And no differences in bond strength were found between cp-Ti and Co-Cr alloy.

Key Words

Shear bond strength, Composite resin veneered metal restorations, cp-Ti, Co-Cr alloy

Composite resin-veneered metal restorations can be used as an alternative to a porcelain-fused-metal restorations. The advantages of veneering composite materials include easy handling, excellent esthetics, hardness similar to that of the natural tooth enamel and ability to be readily repaired.¹ However, the chief disadvantage is the relatively low bond strength of veneering composite to metal framework compared with porcelain-fused metal restorations.^{2,3}

The cobalt-chrome (Co-Cr) is a good alternative to the nickel-chromium-beryllium (Ni-Cr-Be) alloy⁴ which can lead to problems of polymetallism and allergic responses. Recent improvements in casting technology have made it possible to accurately fabricate prostheses made from commercially pure titanium (cp-Ti), thus expanding the use of this metal in clinical prosthodontics.

Various surface treatment methods have been introduced to improve the bond strength of composite resin and metal alloy. Until recent years, mechanical retentions such as undercuts, beads, loops, wires have been indispensable. 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 Lavaditis and Thompson⁵, is technique sensitive and only some Nickel-chromium and cobalt-chromium alloys can be successfully etched.⁶ Sandblasting with alumina powder has also been used to clean the alloy surfaces and to achieve both microretentive topography and increased surface area. It results in a highly activated surface which can be shown by the increased wettability of this material.^{7,8}

Chemical bonding systems are divided into two categories, according to chemical adhesion on metal framework: (1) surface modification techniques which create thin layers of inorganic,

organic, metallic, or composite material on the substrate metal alloys and (2) the use of metal conditioner including functional monomer for targeting direct chemical bonding between metal elements on the alloy surfaces and resin components in the opaque resins.

The Silicoater system (Kulzer Co., Friedrichshof, Germany) was introduced in 1984 by Tiller and Musil⁹ claiming that it was the first system to provide chemical metal-resin bonding. In the silicoating system, a silica layer is pyrolytically applied to the surface over in which a silane coupling agent is applied. The Silicoater system has been substantially modified and released as the Silicoater MD¹⁰ and Kevloc¹¹ systems. The Rocatec system have been developed with is further refinements and simplifications of the original "flame-sprayed" method. Kenneth et al.¹² demonstrated that this technique provided a stronger bond of composite to metal than mechanical retention alone. Others have also demonstrated the efficacy of the silicoating technique.¹³⁻¹⁵

The Metal Primer II bonding system (GC Co., Tokyo, Japan) followed the first group containing functional monomers of MEPS (methacryloyloxyalkyl thiophosphate derivatives), which react chemically with metal alloys. The layer of bonding agent was provided with a consistency of paste and applied on a sandblasted alloy surface and finally polymerized.

Several studies have compared bond strengths with metal alloys of metal conditioner systems and the silicoating systems. Fujishima et al.¹⁶ determined bond strength to cp Ti and Co-Cr alloy using Silicoater MD, New Metacolor (Sun Medical Co., Kyoto, Japan), Ceased (Kuraray, Osaka, Japan) systems and achieved strong bond with all the bonding systems with exception of Thermoresin LC II (GC co., Tokyo, Japan), which is designed for noble metals. In the study of Yanagida et al.,¹⁷ surface treatment with Ceased Opaque

Primer II and Siloc system exhibited significantly greater bond strength than other seven metal conditioners.

The purpose of this study was to compare the shear bond strength of different combinations of two resin bonding systems and two composite veneering resins to cp-Ti and Co-Cr alloy.

MATERIAL AND METHODS

Preparation of cast metal plate

The test procedures were performed according to ISO 10477⁸. The materials used in this study are summarized in Table I-III.

Rectangular specimens with length of 20 mm, width of 10 mm and thickness of 2 mm were constructed. The titanium specimens consisted of titanium grade II (99.4 wt percentage titanium, 0.11 wt percentage oxygen, 0.04 wt percentage iron) and cobalt-chromium specimens consisted of 65 wt percentage cobalt, 27.7 wt percentage chromium, 5.5 wt percentage molybdenum. They were cast from the alloy according to the manufacturers' specifications and then all specimens were wet-

ground (Al_2O_3) using a Labopol-1 grinding machine (Struers, Denmark) to achieve a coplanar surface.

Preparation of specimens for shear testing

The specimens were divided into four experimental groups of five specimens according to the combinations of two bonding systems and two resins to cp-Ti / Co-Cr alloy as listed in Table IV. MG and MS groups were blasted $50\mu m$ Al_2O_3 for 10 s with a sandblaster (Basic master, Renfert GmbH, Hilzingen, Germany). Air pressure was 2 bar and the distance of the nozzle from the metal surface was approximately 10mm. And then, Metal primer II was applied and dried for 60 s. RG and RS groups were blasted-clean (10 s) with $110\mu m$ Al_2O_3 corundum (Rocatec Pre; 3M ESPE, Seefeld, Germany) at a pressure of 2.8 bar. In the second step, the surface was tribochemically coated using Rocatec Plus (3M ESPE, Seefeld, Germany) at a pressure of 2.8 bar for 13 s. And then, silane coupling agent (3M ESPE Sil; 3M ESPE, Seefeld, Germany) was applied and allowed to dry for 5 min.

Table I. The metals used in this study

Metals	Manufacturer	Component
cp-Ti	MANFREDI Inc, Italia	ASTM grade II, Ti 99.4%
Co-Cr alloy	Ticonim D/V Inc, America	Co 64%, Cr 27.7%, Mo 5.5%

Table II. The resin bonding systems used in this study

Trade name	Manufacturer	Component
Metal Primer II	GC Corp., Tokyo, Japan	MEPS* in methyl methacrylate
Rocatec system	3M ESPE, Seefeld, Germany	Rocatec-Pre, Rocatec Plus, 3M ESPE Sil

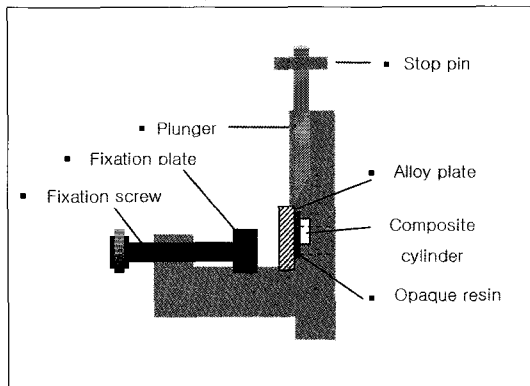
*MEPS = methacryloyloxyalkyl thiophosphate derivative.

Table III. The composite veneering resins used in this study

Trade name	Manufacturer	Component		Type
Gradia	GC Co., Tokyo, Japan	Bis(methacryloyloxy)-propoxy-	5.6wt%	Opaque (paste) Dentin (paste)
		carbonylaminohexane-triazine-trione	26.7wt%	
		di-carbamate	9.9wt%	
		dimethacrylate	46.7wt%	
		aluminoborosilicate glass	10.8wt%	
SiO ₂	0.3wt%			
Sinfony	3M ESPE, Seefeld, Germany	Bis-(meth)acrylate, polyalkenoate	45.3wt%	Opaque (powder/liquid) Dentin (paste)
		silinated SiO ₂ , glass filler	54wt%	
		catalysts, pigments	0.4-0.7wt%	

Table IV. Experimental design

Metals	Code	Testing combinations
cp-Ti / Co-Cr	MG	Sandblasting + Metal PrimerII + Gradia
	MS	Sandblasting + Metal PrimerII + Sinfony
	RG	Rocatec System + Gradia
	RS	Rocatec System + Sinfony

**Fig. 1.** Assembly for shear bond testing.

After surface preparation, composite veneering resin was bonded to the alloy. MG and RG groups: a thin layer of Gradia opaquer was applied twice and light-cured for 30s each with the SPEKTRA LED (Schutz Dental DmbH, Rosbach,

Germany). Thus, a brass ring with a height of 2.5mm and a diameter of 5mm was placed over the opaquer surface. The veneering composite was layered in two steps and every layer was light-cured for 30s and post-curing was performed for 3 min. MS and RS groups : Two layers of Sinfony opaquer were coated and light-cured (2 × 5 s) using the ESPE Visio Alpha device (3M ESPE, Seefeld, Germany). The veneer composite Sinfony was polymerized onto the opaquer layer in two steps and every layer was light-cured for 10 s and finally cured in vacuum in the Visio Beta device (3M ESPE, Seefeld, Germany).

Measurement of shear bond strength

Shear bond strength tests were performed with a universal testing machine (Zwick Z010, Ulm, Germany) with a pushing device used at a crosshead speed of 1mm/min until fracture (Fig.

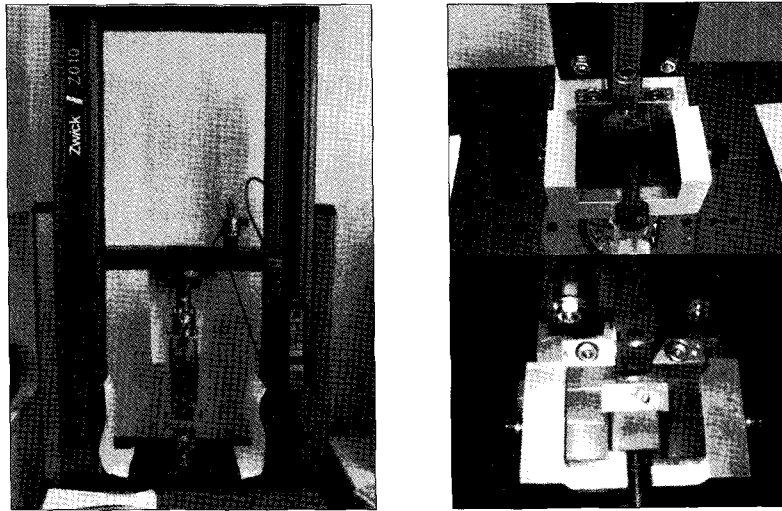


Fig. 2. Universal testing machine (Zwick Z010, Ulm, Germany).

1 and 2). 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).

Statistical analysis

Mean value and standard deviation were computed in each experimental group. Statistical analysis of the results was carried out with Mann-Whitney test and Kruskal-Wallis test with the SPSS for Windows Software (SPSS 13.0; SPSS Inc., Chicago, IL, U.S.A) at the significance level of 0.05.

RESULTS

The bond strength values for the different alloys, bonding systems, and composite veneering resins are summarized in Table V and Fig. 3. Fig. 3 presents the mean bond strength cp-Ti versus Co-Cr alloy.

All tested veneer resins and bonding systems passed the requirements of ISO 10477 (>5 MPa) for bonding to cp-Ti and Co-Cr alloy.

The mean (standard deviations) of shear bond

strength of cp Ti ranged from 16.44 MPa (1.42) to 17.07 MPa (1.69). There were no significant differences between Metal Primer II and Rocatec system to cp-Ti.

The mean (standard deviations) of shear bond strength of Co-Cr alloy ranged from 16.26 MPa (1.46) to 17.70 Mpa (1.66). There were no significant differences between Metal Primer II and Rocatec system to Co-Cr alloy too.

The Mann-Whitney test revealed that the variance among the four groups (MG, MS, RG, RS) of each alloys were not statistically significantly different ($p > 0.05$). And Kruskal-Wallis test demonstrated no significant difference between cp-Ti and Co-Cr alloy ($p > 0.05$).

DISCUSSION

The new generation of light-activated laboratory resins was developed. These composites contain approximately 66% inorganic fillers and 33% resins matrix by volume, whereas the former generation of composites contained 33% inorganic fillers. Change in the size and distribution

Table V. Mean shear bond strengths (MPa) and standard deviations (SD) of composite veneering resins to cp-Ti and Co-Cr using two bonding systems

Groups	cp-Ti		Co-Cr	
	Mean	SD	Mean	SD
MG	16.71	2.03	17.70	1.66
MS	16.44	1.42	16.26	1.46
RG	17.07	1.69	16.61	1.06
RS	16.63	1.96	17.63	0.61

of the filler particles, significantly improved these new composites.^{19,20} They are used in veneered crowns (partial or complete veneers), retainers and pontics for resin-bonded fixed partial dentures, and overlay removable partial dentures and implant prosthesis.^{21,22}

Although base metal alloys have been developed for dental restorations, a nickel-chromium-beryllium (Ni-Cr-Be) alloy may be undesirable because of reported Ni sensitivity in the population.²³ There is also concern about the use of Be in dental alloys because of its potential carcinogenicity.²⁴ To eliminate the potential health hazards of Ni and Be, Co-Cr alloys with comparable physical properties are an alternative.

The use of titanium for cast restorations and fixed partial dentures has increased substantially in recent years, mainly because of the development of new generation of casting machines and investment materials. Further, titanium has many advantages as a dental prosthesis material, including excellent biocompatibility, good mechanical properties and low density,¹⁶ corrosion resistance and improved wear resistance.²⁵ Also, due to the passive oxide film which forms on its surface, resin adheres to cp Ti as effectively as it adheres to other non-noble dental alloys.²⁶ Demand is increasing for resin-veneered crowns, such as those used in telescopic prostheses like a Konus Krone-type crown, as well as those used for

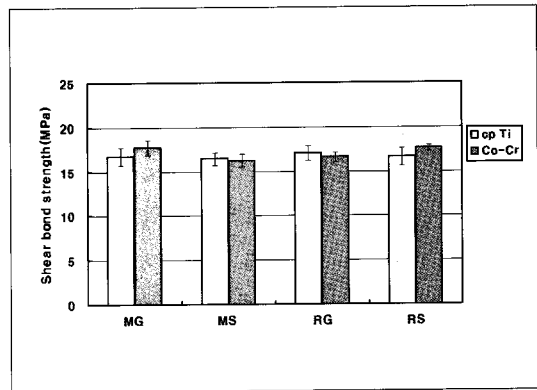


Fig. 3. Graphic representation of bond strength values of all groups.

fixed prostheses for implants. Titanium has excellent characteristics for these purposes.

Although cp-Ti and Co-Cr alloy have the advantages over other base metal alloys, several problems worthy of attention have been reported. One of these is the insufficient bonding between veneering resins and metal framework. Most debonding of resin-bonded prostheses is due to failure at the resin/metal interfaces.²⁷ The factors contributing to this failure are: the type of adhesive and the nature of the alloys, the treatment of the surfaces and the stresses undergone by the prosthesis.²⁸

Several methods available for bonding resin veneers to cast prostheses have been developed. Although mechanical retentions may produce high bond strength²⁹, it can result in microleakage and require high thickness of material necessitating overcontouring of restorations.³⁰ Since the 1980s, methods have been reported to create a chemical bond between dental alloys and composites. The chemical attachment of an opaque layer to metal surface limited microleakage at the resin-metal interface³¹ that occurred because of polymerization shrinkage³² and mismatch of coefficient of thermal expansion between the composite and the

metal.³³ Imbery et al.²⁹ found that the chemical bonding of resin to metal required less metal and allowed more space for properly contoured and esthetic veneer resin compared with mechanical retention.

For this study, chemical bonding systems and their corresponding composites were selected to evaluate the effect of different surface preparations (Metal Primer II and Rocatec system) on bonding between composite veneering materials (Gradia and Sinfony) and cp-Ti / Co-Cr alloy. The shear testing results showed that no significant differences in ability to bond strength were found between Metal Primer II and the Rocatec system.

Metal Primer II contains a methacryloyloxyalkyl thiophosphate derivatives (MEPS) together with a radical polymerization initiator in the single liquid composition. Durability of the bond to metal alloy of composite material is considered to be derived from the resistance to detachment the opaque material from the metal surface. Specifically, the MEPS monomer consists of three differently functioning components; methacryloyl, alkyl and thiophosphate groups. Of these, the methacryloyl group is undoubtedly indispensable to copolymerize the MEPS monomers in the primer and the matrix monomers in the opaque material. The thiophosphate group chemically bonds to metal oxides, probably bonding covalently or ionically by condensing dihydrogen oxide, i.e. water. However, the consistency of the chemical bond between the hydrogen phosphates and metal oxides is somewhat limited, especially against hydrolysis in the oral environment. The adhesive monomer therefore should have a hydrophobic component adjacent to the phosphate group to prevent penetration of water into the adhesive interface. Hence the alkyl group of the MEPS monomer forms a barrier to water penetration at the dihydrogen phosphate-metal oxide

interface. Although this bonding mechanism has not been confirmed by spectroscopic studies, bond strength reports support the effectiveness of the MEPS monomer in bonding other base metal alloys, the surfaces of which are coated with thin oxide layers.³⁴⁻³⁶ Imai et al.³⁷ reported that the combinations of adhesive primer containing thiophosphate monomer and resin cement produced durable bond to both noble and base metal alloys. Taria et al.³⁸ demonstrated Ceased Opaque Primer and Metal Primer II were superior to other primers for strong bond to titanium and resin cement.

The Rocatec system consists of Rocatec Pre, Rocatec Plus of Soft, ESPE Sil. In this system, the silicate layer is formed by sandblasting with a 110 μ m silicate-quartz (Rocatec Pre). When sandblasting particles hit metal surfaces, their kinetic energy is transformed to thermal energy, which may reach the melting point of the metal alloy.²⁹ The melting of the metal alloy is limited to 1 or 2 microns from the surface. The tribochemical effect of airborne particle abrasion results in deposition of molecular coating alumina and silicate particles (110 μ m Rocatec Plus or 30 μ m Rocatec Soft) on the alloy surface. A silane solution (3M ESPE Sil) is also applied to the tribochemically treated metal surface as a coupling agent between free bonds of the silicate surface on the metal and ends of organic polymer chains the opaque material.³⁹⁻⁴¹

Different experimental designs have been used in other studies that have compared bond strengths with metal alloys of metal primer systems and the silicoating systems. Stefanos et al.³⁰ used the Rocatec, Silicoater, Silicoater MD and Sebond, Spectra-Link systems for evaluating bond strength of resin to metal bonding systems and demonstrated that metal conditioner were superior to silicoating systems. Michael et al.⁴² used the Rocatec, Targis Link, Metal Primer and Metal Primer II sys-

tems for evaluating bond strength to cp-Ti and Co-Cr alloy and concluded that Targis Link (phosphoric acid ester system) was superior to others for Co-Cr alloy, both silicoating and functional methods were successful for cp-Ti. Ohkubo et al.⁴³ reported that Ceased Opaque Primer and Metal Primer II exhibited greater bond strength to cast Ti and Co-Cr alloy and durability than did Metal Bond and Siloc system.

In this study, the difference between the shear bond strengths of the cp Ti and Co-Cr alloy was not significant ($p > 0.05$). Fujishima et al.⁴⁴ showed the original Silicoater and the 4-META bonding system could mediate strong bonds between cp Ti and resin composite veneers used with an intermediary layer of an opaque resin, and the bond strengths were similar to those obtained with a Co-Cr alloy. Hanson et al.⁴⁵ demonstrated that while there were no difference between the Co-Cr and the Ti, the Co-Cr and Ti alloy showed better retention to resin than did the three gold alloys after silicoating.

Although the bond durability imparted by the single liquid metal conditioner systems need improvement over silicoating systems, the use of metal priming agents offer the following advantages: (a) simple procedure, (b) non-necessity of a proprietary apparatus and (c) reduced cost performance.

With the results of this study the single liquid metal conditioner system can be more effective and clinically available than silicoating system which is a complicated, technique-sensitive, time-consuming method, when non-precious metal surface is planning to be treated with in order to enhance the bond strength of resin-retained fixed prosthesis.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn:

1. The shear bond strengths of composite resins to both cast cp-Ti and Co-Cr alloy were not significantly different between Metal Primer II and Rocatec system ($p > 0.05$).
2. No differences in bond strength were found between a cp-Ti and a Co-Cr alloy.
3. From the results above, the single liquid metal conditioner system should be more effective and available clinically than silicoating system which is complicate, technique-sensitive, time-consuming method, when non-precious metal surface is planning to be treated with in order to enhance the bond strength of resin-retained fixed prosthesis. Future studies should be performed to evaluate the long-term effects of thermal change and water storage on the resin-to-metal bond.

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