

Bond strength of prosthetic composite to primed and silicoated cast titanium

Kenneth S. Kurtz, DDS,^a Ikuya Watanabe, DDS, PhD,^b Chikahiro Ohkubo, DMD, PhD,^c and David Silken, DDS^d

^aInternational Program in Prosthodontics, New York University College of Dentistry, New York, NY, USA, ^bDepartment of Biomaterials Science, Baylor College of Dentistry, Texas A&M University System Health Science Center, Dallas, TX, USA, ^cDepartment of Removable Prosthodontics, Tsurumi University School of Dental Medicine, Yokohama, Japan, and ^dNew York Hospital Queens, Fresh Meadows, NY, USA

Purpose: The aim of this study was to examine the hypothesis that the bond strength of a polymer-glass composite to cast commercially pure titanium (CPTi) can be improved by treating the metal surface with adhesive metal primers or with heat-processed silicoating.

Materials and Methods: Cast commercially pure titanium disks were prepared and the cast surfaces were air-abraded with 250 µm alumina. There was one control group and four test groups having forty specimens. A second group of forty specimens was similarly prepared, and subjected to a thermal cycling regimen. Three metal adhesive primers (Cesead II Opaque Primer, Metal Primer II, and Solidex) or silicoating (Siloc system) were applied to treated titanium surfaces. Two layers of polymer glass composite (Artglass) opaque and 1-mm thick increments each of dentin and enamel composite components were applied and separately light-polymerized following manufacturer's directions. All specimens were stored in 37°C water for 24 hours. Half of the specimens were thermal cycled in water (4 and 60°C) for 20,000 cycles. Shear bond strength for both groups was measured using a universal testing machine at a crosshead speed of 0.5mm/minute. Results (n=8) were statistically analyzed using ANOVA/Duncan's test ($\alpha=0.05$).

Results: Specimens treated with the Cesead II Opaque Primer showed significantly higher ($p<0.05$) shear bond strength both with and without thermal cycling with no significant decrease in values. The other metal primers and silicoating exhibited lower shear bond strengths with and without thermal cycling.

Conclusion: The Cesead II Opaque Primer improved the bond strength and durability of Artglass to cast pure titanium metal discs. (Int Chin J Dent 2005; 5: 12-16.)

Clinical Significance: In vitro application of the MDP phosphate primer (Cesead II Opaque) demonstrated the highest shear bond strength of a light-polymerized laboratory composite material (Artglass) to cast CP titanium and strength values seemed to be unaffected by thermal cycling. For cast CP titanium prostheses, this type of metal primer may improve the durability of the bonded composite veneer-metal interface and may enhance the esthetic longevity of the fabricated prosthesis.

Key Words: bond strength, cast titanium, metal primer, resin composite, silicoating.

Introduction

The Artglass System, a light-polymerized laboratory composite, described by the manufacturer (Heraeus Kulzer, Wehrheim, Germany) as "polymer-glass", was developed as an alternative veneering material for cast fixed and removable prostheses. The Artglass system can be used for many clinical applications including Artglass bonded to metal for tooth or implant-supported prostheses and a variety of indirect restorations, such as laminate veneers, inlays and onlays.^{1,2} The manufacturer claims that this system causes less wear of opposing dentition. With its ability to be repaired intraorally, Artglass veneering resin combined with cast titanium prostheses may offer clinical advantages equivalent to conventional cast prostheses veneered with feldspathic porcelain. A little information is available regarding the bond strength of Artglass veneered to cast gold alloys^{3,4} or titanium^{3,5} and its alloy,⁶ the latter well-established as a biocompatible material in the oral environment.

Adhesive bonding promoters for metal surfaces have been increasingly used in clinical applications in an effort to create stronger bonding between metal and resin. Numerous studies investigated the composite strength

to treated metal surfaces.⁷⁻¹³ One of these methods is the Silicoater technique (Heraeus Kulzer),¹⁰⁻¹³ in which a silica layer is pyrolytically applied to a surface, then silane coupling agents are applied, to which resins will polymerize. The manufacturer of the Silicoater technique developed several systems, including the conventional Silicoater, Silicoater MD, and Kevloc systems. A recent Silicoater technique, the Siloc system, incorporates refinements and simplifications of previous methods. Principally, the heating mechanism is more controlled than in the Kevloc system.

Adhesive metal primers that can be simply applied to metal surfaces to enhance the bond strength of resins to metal have also been studied.¹⁴⁻²⁰ Cesead Opaque Primer containing MDP (10-methacryloyloxydecyl dihydrogen phosphate) was found to provide high bond strength of resin cements or resin composite to Co-Cr alloys and pure titanium.¹⁵⁻¹⁹ Metal Primer II with MEPS (thiophosphate methacryloyloxyalkyl derivatives) was reported to yield strong bond strength of resin cements to both noble and base metal alloys.¹⁷⁻²⁰ Solidex (4-acryloyloxyethyl trimellitate, 4-AET) was widely used to condition base metal alloys.¹⁹

This study examined the hypothesis that the bond strength of a polymer-glass composite to cast commercially pure titanium can be improved by using several adhesive bonding promoters and heat-processed silicoating.

Materials and Methods

Eighty plastic disk-shaped patterns (10 mm in diameter and 2.5 mm thick) were prepared for casting commercially pure titanium (CP Ti). The disk patterns were invested in mold rings with a magnesia-based investment material (Selevest CB, Selec Co., Osaka, Japan). The burn-out procedure was accomplished according to the manufacturer's instructions and the patterns were then cast with CP Ti (ASTM grade 2, Titanium Industries, Dallas, TX, USA) using a centrifugal titanium casting machine (Ticast Super R, Selec Co.). After casting, the disc surfaces were polished with SiC abrasive paper (No. 600). They were then uniformly air-abraded with 250 μm -sized alumina (Al_2O_3) perpendicular to the surface from a distance of 10 mm at 300 kPa for 10 s with a single use reservoir of alumina followed by ultrasonic cleaning with ethyl alcohol. The surfaces were then treated with the metal primers or silicoating. Unprimed disk specimens were prepared as controls. The primers used in this study were: a primer containing MDP (Cesead II Opaque Primer, Kuraray Co Ltd, Kurashiki, Japan), a metal primer (Metal Primer II, GC Co Ltd, Tokyo, Japan) with MEPS monomer and a metal photo primer (Solidex, Shofu, Kyoto, Japan) based on 4-AET. The silicoating system (Siloc, Heraeus Kulzer) was also applied on the cast titanium surface for comparison with the primers. The silane bonding agent applied to the silicoated surface was Siloc Bond, a proprietary silane coupling agent (3-methacryloxypropyl trimethoxysilane).

After application of the primers and the Siloc system on the sandblasted surfaces, a piece of sticky tape with a circular hole 4.76 mm in diameter was set on the center of the cast disc surface to define the bonding area. Two layers of Artglass opaque resin (Heraeus Kulzer) approximately 100 μm thick each were applied within the hole, and each layer light-polymerized for 90 s with a xenon stroboscopic unit (Uni XS, Kulzer). A Teflon matrix ring (5 mm I.D., 1 mm height) was fixed to the sticky tape around the 4.76 mm diameter area of opaque resin. The ring was filled with Artglass dentin resin and light polymerized for 180 s. The Teflon matrix ring was replaced with a 2 mm height ring, and the enamel resin component was applied atop the dentin layer and light polymerized for 180 s. All polymerization procedures were performed according to manufacturer's instructions. After the removal of the Teflon matrices, all specimens were immersed in 37°C water for 24 hours. This state

was defined as 0 thermocycles (TC 0 - Baseline). Half of the specimens were then subjected to thermocycling, alternating between 4°C and 60°C water for 20,000 cycles (TC 20,000) with a dwell time of 1 minute. Following this treatment, composite-to-metal shear bond strengths were determined for all groups using a universal testing machine (Model 1125; Instron Corp, Canton, Mass, USA) at a crosshead speed of 0.5 mm/minute. Specimen groups were tested prior to and subsequent to thermocycling. For each experimental condition, eight specimens were tested, and the means and standard deviations of shear bond strengths were calculated. The results were statistically analyzed by ANOVA/Duncan's multiple range test ($\alpha=0.05$).

Results

Table 1 shows the results of shear bond strengths. Prior to thermocycling, all the surface-treated specimens showed higher baseline shear bond strength values compared with the control specimens. There were no statistical differences among the control, the Solidex and Siloc specimens. The Cesead II Opaque Primer and the Metal Primer II specimens showed significantly ($p<0.05$) higher shear bond strengths at the baseline than did the other groups. After 20,000 thermocycles, the Cesead II Opaque Primer and the Siloc specimens exhibited decreases in shear bond strength of 15% and 25%, respectively. There were no statistical differences in shear bond strength between the Metal Primer II and the Siloc specimens after 20,000 thermocycles. All control specimens delaminated during thermocycling of 20,000 cycles (100% decrease). The Cesead II Opaque Primer specimens demonstrated the greatest shear bond strength of Artglass to the cast titanium surfaces both before and after thermocycling.

Table 1. Shear bond strength (MPa) of resin bonded to conditioned metal surfaces.

Thermocycle	Control (No treatment)	Cesead II Opaque (MDP-based)	Metal Primer II (MEPS-based)	Solidex (4-AET-based)	Siloc (Silocoater)
0 (Baseline)	21.3 (2.4) b	36.7 (6.0) a	32.3 (4.5) a	24.2 (2.7) b	26.2 (1.3) b
20,000	0	31.1 (3.6)	18.2 (3.6) c	8.7 (7.7)	19.7 (5.6) c

Mean (SD). Similar lower case letters indicate statistically equivalent values (Duncan's test, $p>0.05$).

Discussion

The greatest shear bond strength occurred for Cesead II Opaque Primer before and after thermocycling (Table 1), thus, the primers and silicoating did not exhibit equivalent bond strengths. The Cesead II Opaque Primer contains a hydrophobic phosphate functional monomer (MDP) that strongly bonds the surface oxide layers of base metals such as titanium and cobalt-chromium alloy.¹⁴⁻¹⁸ When Metal Primer II was applied on the cast titanium surface, the bond strength value was similar to that of Cesead II Opaque Primer prior to thermocycling. However, following thermocycling the bond strength for Metal Primer II was lower compared to that of the Cesead II Opaque Primer. Metal Primer II contains MEPS derivatives, which were reported to produce strong bonding of adhesive resin composite to both noble and base metal alloys.¹⁶⁻¹⁹ These results suggest that the MDP-based monomer was superior to the MEPS material for bonding cast pure titanium in this study. Although Solidex Metal Photo Primer contains 4-AET and is widely used to condition base metal alloys, specimens treated with this product showed the lowest bond strength pre- and post-thermocycling. This result suggested that a carboxylic 4-AET derivative is less effective on titanium compared to the MDP and MEPS monomers.

Specimens treated with the Siloc system had bond strengths similar to those when using the Solidex (4-AET) system prior to thermocycling. However, after thermocycling, strength of silicoated metal was greater than that of the Solidex specimens and closed to that of the specimens primed with Cesead II Opaque Primer. The long-term bonding durability of various metal primers and the Siloc silicoating system has also been reported by Yanagida et al.^{5,6} The results obtained in the present study were in agreement with those in their study⁵ conducted using cast Ti and Artglass even though they employed different particle sizes of alumina to air-abrade the metal surfaces. In one of their studies,⁶ the shear bond strength of Artglass to Ti-6Al-7Nb using several types of metal primers (including the Cesead II Opaque Primer) and the Siloc system was tested. They reported that the highest bond strength of Artglass to cast Ti-6Al-7Nb alloy was obtained for the silicoating system, followed by the Cesead II Opaque Primer, showing lower value. In the present study, however, the silicoated specimens did not reach bond strength values similar to those found for the Cesead II Opaque Primer specimens. These differences may be attributed to air abrasion conditions before priming and silicoater application. The size of alumina particles in the present study was 250 μm , whereas they employed 50 μm particles on specimens treated with metal primers and 125 μm particles on silicoated specimens. In the previous study,³ the effect of alumina particle size on shear bond strength of Artglass to silicoated cast titanium and gold alloys has been reported and it was found that using coarser alumina particles increased surface roughness, resulting in increased shear bond strength. Thus, the present study postulates that if 125 μm alumina had been applied to all metal primed surfaces in the study by Yanagida et al.,⁶ the highest shear bond strength might have been obtained for the Cesead II Opaque Primer for Ti-6Al-7Nb.

Conclusion

Of the metal primers employed in this study, the Cesead II Opaque Primer produced the greatest bond strength both pre-and post-thermocycling of a polymer-glass composite (Artglass) to cast pure titanium metal that had been air-abraded with 250 μm alumina particles.

Acknowledgment

Editorial assistance by Mrs. Jeanne Santa Cruz is appreciated.

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Reprint request to:

Dr. Ikuya Watanabe
Department of Biomaterials Science, Baylor College of Dentistry
Texas A&M University System Health Science Center
3302 Gaston Ave., Dallas, TX 75246, USA
Fax: +1-214-828-8458 E-mail: iwatanabe@bcd.tamhsc.edu

Received December 6, 2004. Revised December 16, 2004. Accepted December 23, 2004.
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