Bonding Indirect Resin Composites to Metal: Part 2. Effect of Alloy Surface Treatment on Elemental Composition of Alloy and Bond Strength

***Haralampos Petridis, DDS, MS, PhD*[a]**
***Pavlos Garefis, DDS, PhD*[b]**
***Hiroshi Hirayama, DDS, DMD, MS*[c]**
***Nikolaos M. Kafantaris, DDS, PhD*[d]**
***Petros T. Koidis, DDS, MS, PhD*[e]**

Purpose: This laboratory study compared the effect of different surface treatments of a medium-gold, high-noble alloy on the shear bond strength of an indirect, highly filled resin composite to the alloy and on the elemental composition of the alloy surface. **Materials and Methods:** Ninety disks, cast in a medium-gold, high-noble porcelain-fused-to-metal alloy (V-Deltaloy), received three different surface treatments: sandblasting with 50-µm Al_2O_3 (group 1) or 250-µm Al_2O_3 (group 2) and chemical agents, or with 250-µm Al_2O_3 without chemical agents (group 3) prior to bonding of an indirect resin composite (Artglass, and chemical agents Siloc-pre and Siloc-bond). The specimens were tested in shear, half of them after 24-hour dry storage at room temperature and the rest after 10-day storage in normal saline solution at 37°C and thermocycling (2,500 cycles between 5 and 55°C). Morphologic and qualitative changes on the alloy surface after sandblasting with 50- or 250-µm Al_2O_3 were examined by SEM using EDS analysis and compared with polished specimens. Statistical analysis was performed using two-factor ANOVA. **Results:** The mean shear bond strengths (in MPa) after dry or wet storage and thermocycling were 29 and 24 for group 1, 21 and 18 for group 2, and 17 and 12 for group 3, respectively; there was a statistically significant difference among the groups. Sandblasting of the alloy surface led to statistically significant changes in elemental composition. These changes were of greater magnitude when 50-µm Al_2O_3 particles were used. **Conclusion:** The particle size used for sandblasting influences the shear bond strength between a high-noble alloy and a highly filled indirect resin composite, as well as the elemental composition of the alloy surface. *Int J Prosthodont 2004;17:77–82.*

A new generation of indirect, highly filled resin composites was introduced to the market in the late 1990s.[1,2] These resin composites are accompanied by metal-resin bonding systems to be used in combination with a metal substructure.[1] Metal-resin bonding techniques have evolved through the years from using mechanical retention to the use of bonding agents for chemical retention. Mechanical retention is achieved by the use of metal beads, mesh and pitted metal (macromechanical retention), or by sandblasting or chemical or electrolytic etching (micromechanical retention). Chemical bonding is achieved either directly with adhesive monomers or indirectly through an interfacial layer.[3–5] Metal primers and silane coupling agents are used to promote chemical bonding between the organic groups of the resin composites and the inorganic alloy surfaces.[6–8]

All novel metal-resin bonding systems use bonding agents that lead to some form of chemical bonding with the different alloys, which augments the micromechanical retention that is provided by sandblasting.[9]

[a]*Lecturer, Department of Fixed Prosthesis and Implant Prosthodontics, School of Dentistry, Aristotle University, Thessaloniki, Greece.*
[b]*Professor and Head, Department of Fixed Prosthesis and Implant Prosthodontics, School of Dentistry, Aristotle University, Thessaloniki, Greece.*
[c]*Professor, Department of Prosthodontics and Operative Dentistry, and Director, Division of Postgraduate Prosthodontics, Tufts University School of Dental Medicine, Boston.*
[d]*Professor, Department of Fixed Prosthesis and Implant Prosthodontics, School of Dentistry, Aristotle University, Thessaloniki, Greece.*
[e]*Associate Professor, Department of Fixed Prosthesis and Implant Prosthodontics, School of Dentistry, Aristotle University, Thessaloniki, Greece.*

Correspondence to: *Dr Haralampos Petridis, 32 Proksenou Koromila Street, Neo Panorama, 55236 Thessaloniki, Greece. e-mail: Lpetridis@the.forthnet.gr*

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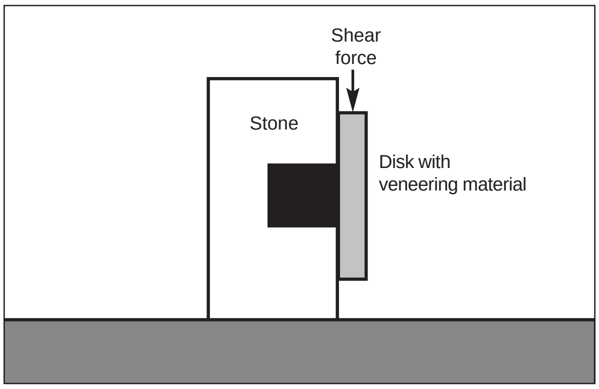


Fig 1 Test design.

Sandblasting of the alloy surface is indispensable in any novel metal-resin bonding technique, as it enhances the bond strength by creating a larger and more "active" surface and by increasing its wettability.[1–4,9–12] Sandblasting is done with aluminum oxide particles of different sizes (usually 37 to 250 μm). The particles move at great speeds, and their collision with the alloy surface leads to a transformation of most of their kinetic energy to heat. The result is a localized meltdown of the alloy, a change in structure, and enrichment in alumina.[10,13–18]

The effects that these qualitative changes have on the bond strength between the alloys and veneering materials have not been studied. The only available papers concern the effect of Al_2O_3 particle size on metal-resin bond strength, but the results are conflicting. Some studies[19–21] have shown that smaller particle sizes lead to higher metal-resin bond strengths, whereas others[12] have shown no difference. The aforementioned studies do not provide an explanation for the differences observed.

The purpose of this laboratory study was to compare the effect of different surface treatments of a medium-gold, high-noble alloy on the elemental consistency (wt%) of the alloy and on its shear bond strength with an indirect, highly filled resin composite. The null hypothesis was that surface treatment would not have any effect on the aforementioned variables.

Materials and Methods

The method of specimen fabrication has been described in detail previously.[22,23] Ninety disks were cast in a medium-gold, high-noble porcelain-fused-to-metal alloy (Au 54.2%, Pd 31.0%, Ag 4.8%, In 9.0%; V-Deltaloy, Metalor Dental). Any irregularities were removed from the cast specimens, and they were gradually polished on flat surfaces with silicon-carbide papers up to 600 grit. The alloy specimens were then steam cleaned and divided into three groups for different alloy surface treatments.

In group 1 (50-μm Al_2O_3 + chemical) and group 2 (250-μm Al_2O_3 + chemical), the metal disks were subjected to 10 seconds of sandblasting using 50- or 250-μm Al_2O_3, respectively, with a pressure of 0.34 MPa and subsequently steam cleaned. They were then treated according to the manufacturer's recommendation for resin bonding with a bonding system that uses an SiO_2 layer and silane coupling agents (Siloc, Heraeus Kulzer). The metal disks received a liberal coating of a primer (Siloc-pre, Heraeus Kulzer) with a single-brush application, which was allowed to dry for 2 minutes, before they were placed in a special preheated oven (Siloc unit, Heraeus Kulzer) under program No. 2 for noble alloys (4 minutes at 250°C). At the end of the cycle, the specimens were removed from the oven and allowed to cool down for 4 minutes. After that, a liberal coat of a bonding agent (Siloc-bond, Heraeus Kulzer) was applied with a single-brush application and allowed to dry for 5 minutes. Finally, three thin layers of opaque and three layers of dentin resin composite (Artglass, Heraeus Kulzer), with a maximum thickness of 2 mm each, were applied. All resin composite layers were applied through a custom Teflon mold cylinder (DuPont) with an internal diameter of 6 mm that delineated the bonding surface of the alloy. The overall thickness of the layers was 5 mm. Each layer was cured in a special unit (UniXS, Heraeus Kulzer) under a strobe light for 90-second cycles, with a final cure of 180 seconds for the whole specimen.

In group 3 (250-μm Al_2O_3 only), the metal disks were subjected to 10 seconds of sandblasting at a pressure of 0.34 MPa using 250-μm Al_2O_3 and subsequently steam cleaned. The opaque and dentin resin composite layers were applied in a similar way to groups 1 and 2, but without the intervention of the metal-resin bonding system.

Half of the specimens of all groups were tested after 24 hours of dry storage at room temperature. The rest of the specimens were stored in 0.1% M NaCl (0.9%) solution at 37°C for 10 days, then thermocycled and tested. The thermocycling procedure was performed in an apparatus that cycled 2,500 times between 5 and 55°C water baths with a dwell time of 20 seconds in each one. It has been shown that this combination of water storage and thermocycling is adequate to give an indication of the condition that occurs in vivo over several years.[24,25] The composite that was bonded to metal was embedded in a type IV stone (Die-Keen, Heraeus Kulzer) in a copper cylinder, with the metal disk parallel to the cross-section of the cylinder (Fig 1),

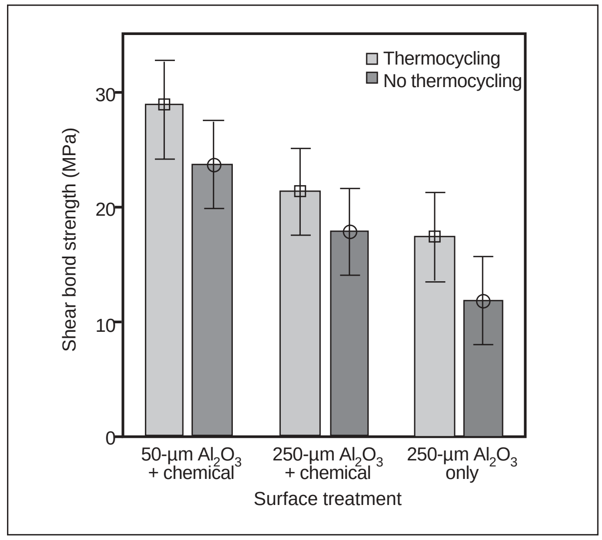
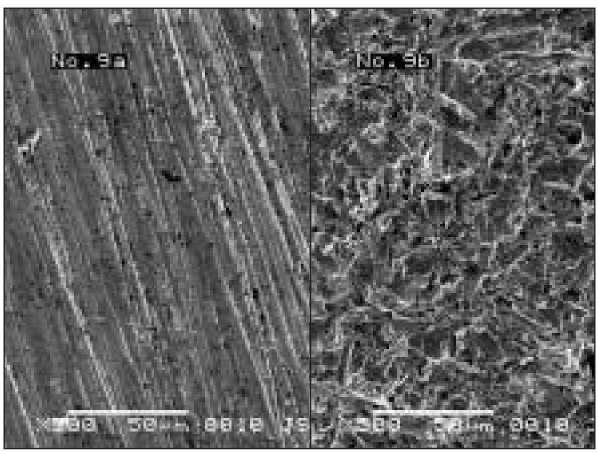
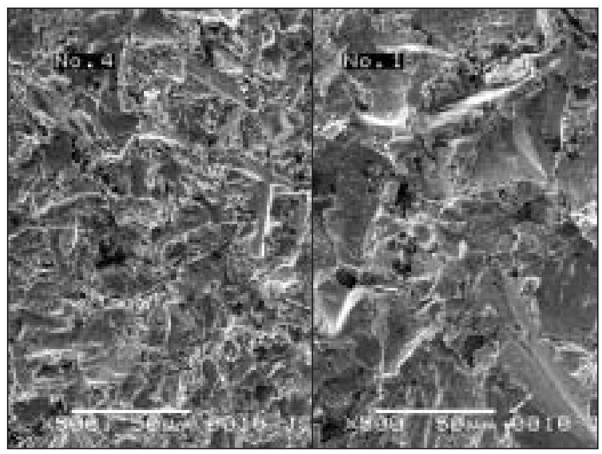


Fig 2 *(right)* Mean shear bond strength values (*error bars* show ± 1.0 standard deviation).

Fig 3 *(below)* SEM analysis: *left* = 600-grit SiO_2; *right* = 50-µm Al_2O_3.

Fig 4 *(below right)* SEM analysis: *left* = 50-µm Al_2O_3; *right* = 250-µm Al_2O_3.





and mounted on a universal testing machine (Instron). The specimens were tested in a parallel shear test, with a cross-head speed of 0.5 mm/min, until breakage occurred.

To compare the morphologic and elemental changes of the alloy surfaces after sandblasting, nine more disks were prepared and divided to receive three surface treatments: polishing with sandpaper up to 600 grit, and sandblasting with 50- or 250-µm Al_2O_3. Scanning electron microscopy (Environmental SEM, JSM-5900 LV, secondary detector, Jeol) was used for optical analysis, with an accelerating potential of 15 kV and 500× enlargement. The wt% elemental composition was measured using energy dispersive spectroscopy (EDS) analysis (Phoenix EDS detector, EDAX) with an accelerating potential of 15 kV. To reduce variability, two measurements of different areas were made for each sample, which gave six measurements for each surface treatment.

Statistical analysis was performed to compare differences in bond strength and elemental consistency, using analysis of variance (ANOVA) to determine significant differences between the groups. To compare differences of bond strength between different bonding techniques before and after thermocycling, a one-way ANOVA was performed, followed by a post hoc Tukey test.

Results

The mean shear bond strength values (in MPa) for the groups tested after dry or wet storage and thermocycling were 29.0 and 23.8 for group 1, 21.4 and 17.9 for group 2, and 17.3 and 11.8 for group 3, respectively (Fig 2).

A Levene test of equality of error variances was performed, and the variance through the three groups was found to be equal, which permitted an ANOVA to be performed. The analysis revealed that the effect of the factors “treatment” and “thermocycling” on the shear bond strength was statistically significant ($P < .001$). The interaction of these two factors was not statistically

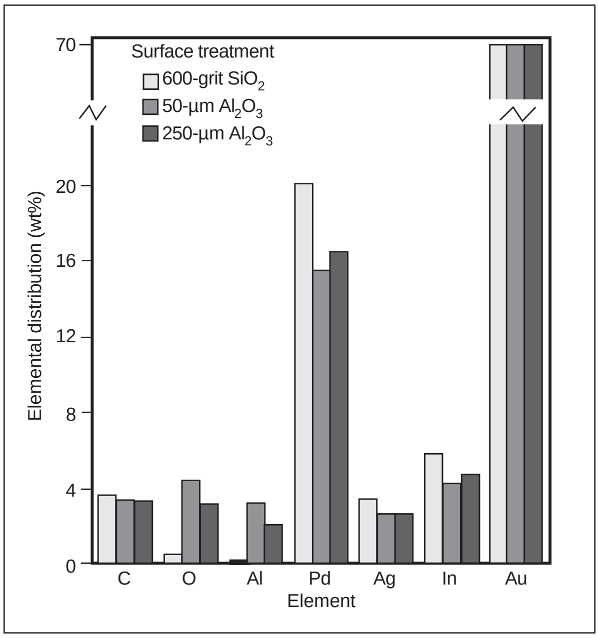
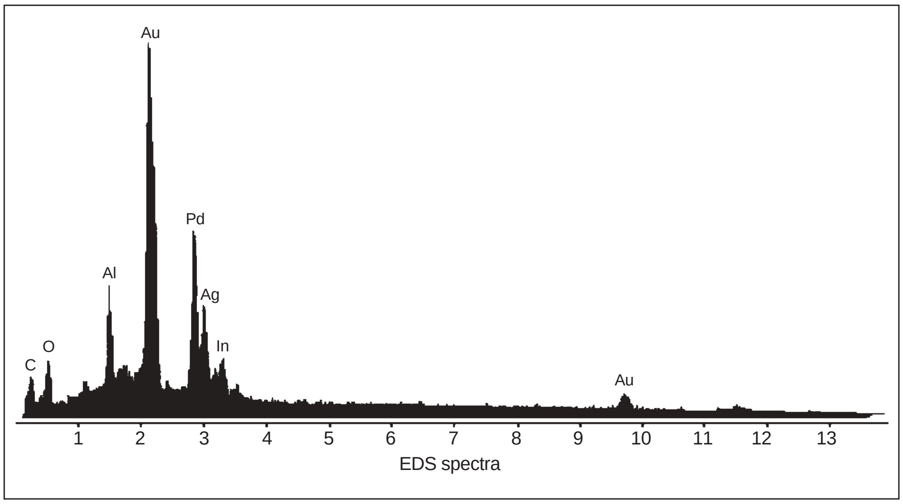


Fig 5 *(left)* Mean changes in elemental composition of the alloy surface after sandblasting.

Fig 6 *(below)* Representative EDS spectra of the alloy surface after sandblasting with 250-µm Al_2O_3 particles.



significant ($P = .521$). Comparison of the three groups revealed that group 1 was the strongest, followed by groups 2 and 3 ($P < .001$), regardless of storage conditions.

The SEM analysis revealed differences in surface morphology among the three treatments. Sandblasting created a rougher surface compared with the 600-grit treatment (Fig 3), and treatment with 50-µm particles created a finer microtopography than treatment with 250-µm particles (Fig 4). ANOVA revealed that the effect of the factors "element" and the interaction of "element" and "treatment" were statistically significant ($P < .001$). This led to a separate analysis for each element to determine the exact changes that occurred. The analysis revealed that sandblasting led to a statistically significant ($P < .001$) increase in the elemental consistency of Al and O and a decrease of Pd, Ag, and In (Figs 5 and 6). The wt% consistency of Au did not change significantly. For all elements except Ag, the change was greater ($P < .050$) for the specimens sandblasted with 50-µm Al_2O_3 compared to 250-µm Al_2O_3.

Discussion

The results of the present study clearly favor the use of particle sizes of 50 µm compared to 250 µm when sandblasting a high-noble alloy for bonding an indirect resin composite. This finding is in agreement with previous studies.[19–21] The literature does not provide any explanation for these findings. One possible explanation in our study is the improved microtopography that is created when sandblasting with 50-μm particles, as can be seen in the SEM analysis, which might have increased the surface wettability and penetration of the bonding agents into the microirregularities. Another possible explanation for the better results achieved when sandblasting with 50-μm particles may be the change in the elemental composition observed.

As expected, sandblasting led to an increase in the quantity of O and Al of the alloy surface, in agreement with previous studies.[15–18] Sandblasting with 50-μm compared with 250-μm particles led to an increase of 43% and 57% in O and Al, respectively. A previous study[17] of the surface of commerically pure titanium after sandblasting reported a smaller increase in the elemental concentration of Al with smaller Al_2O_3 grain size. One possible explanation for the difference observed between grain sizes may be the particle size distribution in the sandblasting powders. The small particle size (50 μm) powders present a narrow particle size distribution of between 47 and 120 μm. The large particle size (250 μm) powders present a wide particle size distribution of between 10 and 700 μm.[17] The smaller particles may be able to better penetrate the alloy surface and be retained.

Sandblasting also led to a drop in the wt% concentration of Pd, Ag, and In compared with the polished surface. This finding is in agreement with the results of a previous study.[18] With the exception of Ag, these changes were greater when 50-μm particles were used for sandblasting compared to 250-μm particles. These elements, especially Al and In, form oxides that are used to bond the interfacial SiO_2 layer in the particular metal-resin bonding technique tested. This significant change of the alloy surface composition after sandblasting might offer a possible explanation for the difference in bond strength values. More research is needed to determine a direct correlation of the two factors.

From the results of the shear bond strength comparison, it can be concluded that the most effective way of bonding the particular indirect resin composite and alloy is by pretreating the metal surface with 50-μm Al_2O_3 particles, followed by the metal-resin bonding system. The group with the lowest shear bond strength values was the group that used only micromechanical retention. Although differences in test design and storage parameters do not permit a direct comparison with other experiments,[26,27] the bond strength values in the present study are in the range reported in similar studies.[20,28,29]

Wet storage and thermocycling led to a statistically significant decrease of shear bond strength values in all groups. This deterioration of strength is in agreement with literature concerning metal-resin bonding[30,31] and has been discussed in a previous article.[22] Dry storage was included in the study to test the effect of chemical bonding on bond stability. Comparison of the bond strengths of groups 2 and 3 showed the enhancement achieved by the addition of chemical bonding. It seems that the addition of chemical bonding reduces the bond degradation in a corrosive environment. The significance of the difference in mean bond strength values between groups 2 and 3 increased from $P = .320$ to $P < .001$ after thermocycling. It must be noted, however, that micromechanical retention seems to account for approximately 80% of the total bond strength before thermocycling and 65% after thermocycling. These findings are in agreement with a previous study.[12]

The results of this laboratory study do not have direct clinical implications, but serve to better understanding of the bond strength dynamics of new metal-resin bonding systems and possibly explain future clinical findings. This study looked at the bond between a noble alloy and an indirect resin composite. More studies are needed to test different alloy compositions and metal-resin bonding systems because the interaction of these factors can significantly alter the findings.[32] Long-term clinical studies should be conducted to validate the use of these indirect resin composites.

Acknowledgments

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Literature Abstract

A retrospective comparison of two definitive impression techniques and their associated post-insertion adjustments in complete denture prosthodontics.

The definitive impression technique is reportedly one of the more critical elements in complete denture prosthodontics. This study compared the number of postinsertion adjustments needed by edentulous patients who had complete dentures fabricated using a modified technique in a retrospective analysis of patients in a private practice setting. Seventy-eight edentulous patients were treated with the traditional technique involving custom light-polymerized impression trays. The impression technique was border molded with modeling plastic impression compound. Another 78 edentulous patients were treated with the modified impression technique involving custom light-polymerized impression trays border molded with heavy polyvinyl siloxane impression material. All definitive impressions were made with light-body polyvinyl siloxane. Patients were followed for 1 year after insertion of complete dentures. Postinsertion adjustments to complete denture flanges were quantified by the number of postinsertion appointments required for 1 year after insertion. The mean number of adjustment visits was 2.68 for both groups; there was no significant difference in the number of adjustments required for patients whose dentures were made with either technique. The author did not conclude that either technique should be taken as the definitive method of fabricating complete denture impressions because of several limitations in the study design.

Drago CJ. *J Prosthodont* 2003;12:192–197. **References:** 30. **Reprints:** Dr Carl J. Drago, Gundersen Lutheran Medical Center, 1836 South Avenue, LaCrosse, Wisconsin 54601. e-mail: cjdrago@gundluth.org—*Myung W. Brian Chang, Lincoln, Nebraska*