

# Bonding Indirect Resin Composites to Metal: Part 1. Comparison of Shear Bond Strengths Between Different Metal-Resin Bonding Systems and a Metal-Ceramic System

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**Purpose:** This laboratory study compared the shear bond strength between three indirect resin composites and a noble alloy using their respective bonding systems. **Materials and Methods:** One hundred twenty disks cast in a medium-gold, high-noble metal-ceramic alloy (V-Deltaoy) were divided equally into four groups and received different treatments for veneering: Conventional feldspathic porcelain (Omega) was applied on one set of specimens to be used as a control, and three indirect resin composites (Artglass, Sculpture, Targis) with their respective bonding systems were used for the other groups. The specimens were tested in a parallel shear test, 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. The fractured specimens were evaluated to determine the nature of the failure. **Results:** The mean shear bond strength values (in MPa), before and after wet storage and thermocycling, were 30 and 23 for the metal-ceramic group, 29 and 23 for the Artglass group, 20 and 19 for the Sculpture group, and 17 and 14 for the Targis group, respectively. The metal-ceramic and Artglass groups exhibited significantly higher bond strengths than the other two groups. All specimens, with the exception of the Sculpture group, showed a significant decrease in bond strength after wet storage and thermocycling. **Conclusion:** No group exceeded the shear bond strength of the metal-ceramic group, but the Artglass group with its respective metal-resin bonding system exhibited similar bond strengths. The Sculpture group showed a stable bond after water storage and thermocycling. *Int J Prosthodont* 2003;16:635-639.

A new generation of indirect, laboratory-fabricated resin composites, so-called “ceromers,” was

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introduced to the market in the late 1990s.<sup>1</sup> These polymers have been proposed for use in similar circumstances as ceramics, with the possible advantages of simpler and less costly fabrication, better wear compatibility against natural teeth, and easier repairs.<sup>2,3</sup>

These indirect resin composites are accompanied by metal-resin bonding systems for use in combination with a metal substructure.<sup>4</sup> The bonding agents lead to some form of chemical bonding with the different alloys, which augments the micromechanical retention provided by sandblasting.<sup>5</sup> There is a lack of studies on the efficiency of most of these metal-resin bonding systems. The technique that has been studied the most in vitro is the interfacial bonding of metals and resin composites through a silicon oxide layer, a technique introduced in 1984.<sup>6</sup> A number of in vitro studies have proven the efficiency of this technique.<sup>7-17</sup> Siloc (Heraeus Kulzer), the bonding system of Artglass (Heraeus Kulzer), uses an SiO layer. A previous study<sup>18</sup> showed that the shear bond strength of Artglass to a noble alloy is significantly lower than the

metal-ceramic bond. In that study, pretreatment of the alloy surface was done using air abrasion with 250- $\mu$ m aluminum oxide particles. Another technique for metal-resin bonding is the ionization of the metal surface. Targis Link (Ivoclar-Vivadent), the bonding system of Targis (Ivoclar-Vivadent), uses this technique. A recent laboratory study<sup>19</sup> found no difference in the probability of failure between a resin-veneered (Targis or Artglass) or metal-ceramic implant-supported restoration. An *in vivo* study<sup>20</sup> comparing the predecessors of Targis Link and Siloc found that use of ionization leads to increased microleakage at the metal-resin interface, while a recent *in vitro* investigation<sup>21</sup> revealed no difference between the techniques.

For clinical success, the veneering material should be strongly bonded to the metal substructure, without interfacial leakage or delamination. Delamination of porcelain from metal in metal-ceramic restorations is extremely low.<sup>22-24</sup> Therefore, the bond strength of porcelain to the metal substrate can be considered the standard against which alternative veneering materials can be tested.

The purpose of this laboratory study was to compare the shear bond strength between a medium-gold, high-noble alloy and three resin composite veneer systems with that between the same alloy and a feldspathic porcelain. The null hypothesis was that there would be no difference in bond strength values between the groups.

### Materials and Methods

One hundred twenty disks (15 mm in diameter and 1.5 mm thick) were cast in a medium-gold, high-noble metal-ceramic alloy (Au 54.2%, Pd 31.0%, Ag 4.8%, In 9.0%; V-Deltaloy, Metalor Dental). The method of specimen fabrication has been described in detail previously.<sup>18</sup> 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, subjected to 10 seconds of sandblasting using 50- $\mu$ m aluminum oxide at 50 psi, and subsequently steam cleaned. The specimens were divided into four groups and received treatments for veneering. All veneering 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.

Group 1 was treated according to the manufacturer's recommendation for conventional feldspathic porcelain application (Omega, Vita). A thin wash layer of opaque was applied, followed by a second opaque layer and two dentin body layers, each fired separately.<sup>18</sup>

In group 2 (Artglass), the metal specimens were treated according to the manufacturer's recommendations for resin bonding. They received a liberal coating of a primer (Siloc-pre, Heraeus Kulzer) with a single brush application; this was allowed to dry for 2 minutes before specimens were placed in a special preheated oven (Siloc unit, Heraeus Kulzer) under program No. 2 for noble alloys. At the end of the cycle, the specimens were removed from the oven and allowed to cool 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 composite (Artglass), with a maximum thickness of 2 mm each, were applied. Each layer was cured in a special unit under a strobe light for 90-second cycles, with a final cure of 180 seconds for the whole specimen.

In group 3 (Targis), a bonding agent (Targis Link) was applied on the metal surfaces and allowed to act for 4 minutes. Two opaque layers were applied through the Teflon jig. Each layer was light cured for 20 seconds (Targis Quick, Ivoclar-Vivadent) and processed for a final cycle in a special curing machine (Targis Power, Ivoclar-Vivadent). A disposable sponge was used to remove the unpolymerized superficial layer of the opaque according to the manufacturer's instructions. Three more dentin layers were added and polymerized in the same way as the opaque layers, to a final height of 5 mm. The specimens were subjected to one final light- and heat-polymerization cycle (Targis Power).

In group 4 (Sculpture, Jeneric/Pentron), a silane coupling agent (Sculpture Metal Coupler, Jeneric/Pentron) was applied on the metal surfaces and allowed to dry for 3 minutes in a special oven (Sculpture curing unit, Jeneric/Pentron). The opaque layer of this system differs because it is heat cured and comes in a powder-liquid form. The opaque was mixed in a 1:2 ratio until a homogeneous consistency was reached. It was then applied to the metal surface and subjected to a curing cycle under heat and vacuum (Sculpture curing unit). A thin layer of liquid resin was applied on the opaque layer and light cured for 1 minute. Three dentin layers were built through the Teflon jig. Each layer was light cured for 1 minute, and the specimens were subjected to a final curing cycle under heat and vacuum (Sculpture curing unit).

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.<sup>25,26</sup>

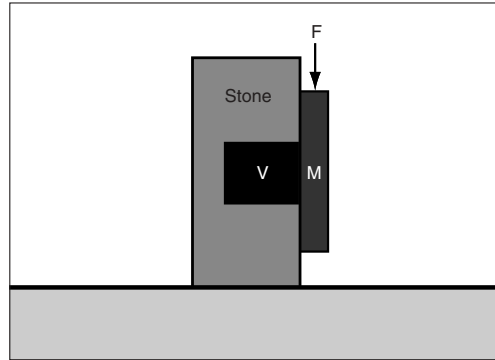
The porcelain or composite that was bonded to metal was embedded in a type IV stone (Die-Keen, Heraeus Kulzer) in a copper tube, with the metal disk parallel to the cross-section of the cylinder (Fig 1), and mounted on a universal testing machine (Instron). The veneering material was supported in dental stone to measure the bond strength of the interfaces, rather than the strength of the material itself. To reduce variability, specimens were checked very carefully to make sure that the veneering material was fully supported by the embedding dental stone. The specimens were tested in a parallel shear test, with a cross-head speed of 0.5 mm/min. The force was applied at the interface of the metal-composite or metal-ceramic until breakage occurred. This design has been shown to produce less variability in the results.<sup>27</sup> The force output from the machine was divided by the bonding surface area, and results were reported in MPa (MN/m<sup>2</sup>). The fractured specimens were evaluated under 10× magnification to determine the nature of the failure (cohesive, adhesive, or combination), as well as the interfaces involved. A failure was described as adhesive if there was an absolutely clean separation of the interfaces.

Levene's test of equality of error variances was performed, and the variance through the four groups was found to be equal, which permitted an analysis of variance (ANOVA) to be performed to determine significant differences between the groups. To compare differences in bond strength between different bonding techniques before or after thermocycling, a one-way ANOVA was performed, followed by a post hoc Tukey test.

## Results

The mean shear bond strength values (in MPa), before and after wet storage and thermocycling, were 29.7 and 22.9 for the metal-ceramic group, 29.0 and 23.1 for the Artglass group, 20.2 and 19.2 for the Sculpture group, and 17.1 and 14.1 for the Targis group, respectively (Fig 2).

ANOVA revealed that the effect of the factors "material" and "thermocycling" on the shear bond strength was statistically significant ( $P = .021$  and  $.05$ , respectively). The interaction of these two factors was also statistically significant ( $P = .006$ ). This means that not all materials behaved the same during thermocycling. There was a statistically significant reduction of the shear bond strength in all groups after thermocycling, with the exception of group 4 (Sculpture).



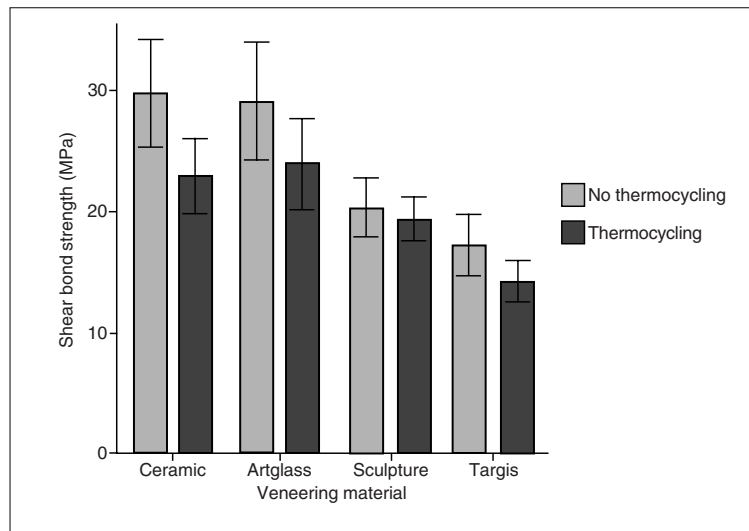
**Fig 1** Test design: metal disk (*M*) with veneering material (*V*) embedded in stone, and the shear force (*F*) acting on the disk.

Comparison of the four groups with the post hoc Tukey test revealed no statistical difference ( $P > .05$ ) in the shear bond strengths of groups 1 and 2, regardless of storage conditions. Both groups exhibited statistically higher bond strengths than groups 3 and 4 ( $P < .001$ ). Groups 3 and 4 showed a significant difference only after thermocycling ( $P < .001$ ), with group 4 having higher bond strength values.

The majority of specimens in groups 1 and 2 presented with combination failure: adhesive (between opaque and metal), and cohesive through the veneering material. Only four specimens presented with adhesive failures. In group 3, all specimens failed adhesively. In group 4, most specimens presented with a combination failure. In six specimens that underwent thermocycling, there was cohesive failure through the opaque layer.

## Discussion

The results of the present study showed that all but one metal-resin bonding system (Artglass) exhibited lower shear bond strength values compared to the metal-ceramic system tested. One issue for consideration is whether metal-resin and metal-ceramic bonds are directly comparable. The test design in our study may have led to an underestimation of the metal-ceramic bond because no compressive forces were developed between the ceramic and alloy after cooling. Furthermore, feldspathic ceramics are much stiffer materials than resin composites (modulus of elasticity for ceramic = 69 GPa, Artglass = 9 GPa, Targis = 10 GPa, Sculpture



**Fig 2** Mean shear bond strength of all test groups (error bars show  $\pm 1.0$  standard deviation).

= 14 GPa).<sup>1,28</sup> To reduce variability caused by the difference in stiffness, specimens were checked very carefully to make sure that the veneering material was fully supported by the embedding dental stone.

To our knowledge, only two published studies have compared novel metal-resin bonding systems with metal-ceramic systems. In a previous study<sup>18</sup> of the same design, the bonding system of Artglass did not compare favorably with the metal-ceramic bond. The difference in the present study was the preparation of the alloy surface, which was sandblasted with aluminum oxide particles of 50  $\mu\text{m}$  (instead of 250  $\mu\text{m}$ , as in the previous study). The effect of alloy surface treatment on the bond strength will be the topic of the second part of this article. A recent laboratory study<sup>19</sup> showed no difference in the probability of mechanical failure (compressive load) between three different types of indirect resin-veneered and one metal-ceramic implant restoration that was used as a control. Two of the systems tested were identical to those in our study (Artglass and Targis). Although the results of that study are not directly comparable to the present study because of differences in experimental design (different test modes and specimen design), the results of the Artglass group agree with our findings. That study<sup>19</sup> also used macromechanical retention of the metal framework in the form of retention beads, which might explain the better results regarding the Targis group.

Wet storage and thermocycling led to a statistically significant decrease of shear bond strength values in all groups except group 4 (Sculpture). This

deterioration of strength is in agreement with the literature on metal-ceramic<sup>29-31</sup> and metal-resin bonding<sup>32,33</sup> and has been discussed in a previous article.<sup>18</sup> The stability of the bond strength values after thermocycling in group 4 (Sculpture) cannot be definitively explained. The technical difference of this system compared with the rest is the fact that the opaque layer is heat polymerized. According to the manufacturer, light-induced polymerization of an opaque layer can be inadequate, whereas heat polymerization can increase the degree of conversion and strength of this critical layer that bonds to the alloy surface. Further research is needed in that regard.

There was no clear correlation between the mode of failure and bond strength values. The weakest group (Targis) experienced only adhesive failures, whereas the majority of failures in the other groups were a combination of adhesive and cohesive. These findings are in agreement with the literature.<sup>16,34,35</sup> One would expect to see a difference in the mode of failure between the Sculpture and Artglass groups because of strength differences of the opaque layer. Such a difference was not noted in this study.

Considering the limitations of any laboratory study, the results of the present study should be viewed with caution regarding clinical significance. An abundance of variables affect clinical success and longevity of veneering materials. The current literature has limited information and no clinical studies regarding the efficiency of indirect resin composites and their metal-resin bonding systems. Therefore, a number of

properties of these materials must be tested in a laboratory environment before clinical studies are initiated. Shear bond strength is a variable that has been tested extensively in metal-ceramic systems. The present findings are valid for the particular alloy and techniques used. The combination of indirect resin composites and their respective bonding systems, as recommended by the manufacturers, was chosen to avoid chemical incompatibilities. Another point that needs consideration is the variability in metal-resin bond strength values that may occur in a commercial laboratory setting because of technique sensitivity. The minimum bond strength that will suffice for clinical longevity has not been established. Comparisons of different systems are helpful in that perspective. These materials and techniques should be tested in vivo in long-term studies for more valid information.

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