Bonding of an indirect composite material to a magnetic stainless steel alloy

Hiroshi Shimizu, DDS, PhD, Tomohiro Kawaguchi, DDS, PhD, Kazuaki Takahashi, DDS, and Yutaka Takahashi, DDS, PhD

Division of Removable Prosthodontics, Department of Oral Rehabilitation, Fukuoka Dental College, Fukuoka, Japan

Purpose: The purpose of the present study was to investigate the bond strength of an indirect composite material to a magnetic stainless steel alloy.

Materials and Methods: The surfaces of machined magnetic stainless steel alloy specimens were abraded with 600-grit silicon carbide paper and then grouped according to the type of surface treatment given: 1) without preparation, 2) tribochemical silica coating, 3) air abrasion with 50 µm alumina, 4) tribochemical silica coating and application of opaque resin, 5) air abrasion and application of a metal conditioner for base metal alloys, and 6) air abrasion, application of a metal conditioner and opaque resin. An indirect composite material (Estenia C&B) was then applied to the treated surfaces of the alloy and polymerized. The specimens in groups 2-6 were thermocycled up to 10,000 cycles. The specimens in groups 5 and 6 were thermocycled up to 20,000 cycles. The shear bond strengths were determined using a universal testing machine.

Results: All of the cylindrical columns of the indirect composite material in group 1 debonded before their shear bond strengths could be determined. Group 6 exhibited the greatest bond strength, followed by group 5, compared to the other groups before thermocycling (p<0.05). The bond strengths of groups 3-6 did not decrease after thermocycling (p>0.05), whereas those of group 2 significantly decreased (p<0.05).

Conclusion: Significant improvement in the bond durability of an indirect composite material to a magnetic stainless steel alloy was achieved by alumina air abrasion and the application of a metal conditioner for base metal alloys and opaque resin, followed by pre-polymerization. (Asian Pac J Dent 2012; 12: 27-31.)

Key Words: alumina air abrasion, indirect composite material, magnetic stainless steel alloy, metal conditioner

Introduction

Dental magnetic attachments nowadays have a clinically sufficient attractive force and have been reduced to an acceptably small size for clinical use. Magfit EX (Aichi Steel Corp., Tokai, Japan)\(^1\) is one of the attachments containing a neodymium-iron-boron magnet in the yoke that can close a magnetic circuit. A magnetizable chromium-rich stainless steel alloy was specially designed to resist corrosion in the outer component of the yoke and the keeper body of this attachment.\(^2\) A conventional cast-bonding technique has been used to fabricate the copings with the keepers. However, it is well known that the keeper surfaces bulge in the center during the casting process, which causes a significant decrease in the attractive force of the cast-bonded keeper compared to its original attractive force and that of the direct-bonded keeper.\(^3\) In the direct-bonding method, careful attention must be paid to polishing the keeper surfaces to recover the attractive force.\(^4\)

New procedures have been developed for a resin-based composite material coping with a keeper\(^5\) in which the resin-based material coping portion is attached to a fiber-reinforced post.\(^6\) Such new non-casting methods may prevent the decrease of attractive force if the keeper and the resin-based composite material are strongly attached to each other. Important improvements in the bond strengths of autopolymerizing acrylic resins to magnetic stainless steel alloys have been achieved.\(^7\)\(^-\)\(^11\) However, little information is available regarding the bond strengths of resin-based composite materials to stainless steel alloys. The purpose of the present study was to evaluate the effect of surface treatments on the shear bond strength of indirect composite material applied to a magnetizable chromium-rich stainless steel alloy. The ultimate goal of this study was to establish the fabrication technique using an indirect composite material coping with keepers.
Materials and Methods

A total of 130 quadrangular-shaped specimens (10x10x1.0 mm) were machined from a magnetizable chromium-rich stainless steel alloy (AUM 20 steel alloy, Aichi Steel Corp.; SUS 316L) and embedded in an autopolymerizing resin material with an acrylic ring. The alloy surfaces were abraded with 600-grit silicon carbide paper under running water. The freshly abraded alloy specimens were divided into six groups according to the type of surface treatment they would receive: 1) without preparation; 2) tribochemical silica coating using the Rocatec system (3M ESPE AG, Dental Products, Seefeld, Germany) with 110 µm grain-sized alumina (Rocatec Pre, 3M ESPE AG, Dental Products), 110 µm grain-sized alumina coated with silicon dioxide (Rocatec Plus, 3M ESPE AG, Dental Products) and a silane coupling agent (Rocatec ESPE-Sil, 3M ESPE AG, Dental Products); 3) air abrasion with 50 µm grain-sized alumina (Aluminous Powder WA 360, Pana Heraeus Dental Inc., Osaka, Japan) for 10 s using a grit blaster (Duostar Z2, Bego, Bremen, Germany). The emission pressure was 0.3 MPa with the nozzle positioned at a right angle approximately 5 mm from the surface of the alloy specimen; 4) tribochemical silica coating followed by the application of opaque resin (Estenia C&B body opaque OA 3.5, Kuraray Medical Inc., Tokyo, Japan) and then polymerized with a light-polymerizing unit (Twinkle L II, Toho Dental Product Co., Ltd., Saitama, Japan) for 3 minutes; 5) air abrasion and priming with a metal conditioner (Estenia C&B Opaque Primer, Kuraray Medical Inc.); and 6) air abrasion, priming with a metal conditioner and application of opaque resin, followed by pre-polymerization. The experimental groups and surface treatments are summarized in Table 1.

Table 1. Surface treatment

<table>
<thead>
<tr>
<th>Group</th>
<th>Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Without preparation</td>
</tr>
<tr>
<td>2</td>
<td>Tribochemical silica coating</td>
</tr>
<tr>
<td>3</td>
<td>Air abrasion with 50 µm alumina</td>
</tr>
<tr>
<td>4</td>
<td>Tribochemical silica coating and opaque resin</td>
</tr>
<tr>
<td>5</td>
<td>Air abrasion and application of a metal conditioner</td>
</tr>
<tr>
<td>6</td>
<td>Air abrasion, application of a metal conditioner and opaque resin</td>
</tr>
</tbody>
</table>

A piece of masking tape with a 6.0 mm diameter hole punched in it was placed on the bonding surface of each specimen, and a Teflon ring with a circular hole (5.0 mm inner diameter and 6.0 mm outer diameter) was placed within the hole in the masking tape to hold it in place and define the bonding area. An indirect composite material (Estenia C&B E1, Kuraray Medical Inc.) was applied inside the Teflon ring and then polymerized with the same light-polymerizing unit for 3 minutes. All the specimens were immersed in water at 37°C for 24 hours. Sixty of these specimens in groups 1-6 referred to as “thermocycling 0” were tested for shear bond strength. Fifty specimens in groups 2-6 were placed in a thermocycling apparatus (Thermal Shock Tester TTS 1, Thomas Kagaku Co. Ltd., Tokyo, Japan) and cycled in water (10,000 cycles) between 5°C and 55°C with a dwell time of 1 minute at each temperature. The remaining 20 specimens of groups 5 and 6 were also cycled up to 20,000 cycles. The shear bond strengths were determined using a universal testing machine (Autograph AGS-J, Shimadzu Corp., Kyoto, Japan) at a crosshead speed of 0.5 mm/minute. The failure mode was evaluated after the shear bond strength testing by examining the fracture surfaces using a magnifying glass with an electric light at 40 x magnification. Failure was classified as adhesive failure at the metal-resin interface, cohesive failure...
within the resin, or a mixture of both adhesive and cohesive failure. The means and standard deviations for the shear bond strengths \((n=10)\) were calculated. The values were statistically analyzed using a one-way analysis of variance (ANOVA) and Newman-Keuls post-hoc comparisons test at a significance level of \(p<0.05\).

**Results**

All of the cylindrical columns of the indirect composite material without preparation debonded prior to determining the shear bond strengths, with no exceptions. Therefore, measurements were not made for this experimental group, and the statistical analysis was carried out without group 1. The ANOVA and Newman-Keuls post-hoc comparisons test indicated that the shear bond strengths of group 6 were the greatest, followed by group 5 both before and after thermocycling. There were no significant differences \((p>0.05)\) in the bond strength between 10,000 and 20,000 thermocycles in groups 5 and 6. There were no significant differences \((p>0.05)\) in the bond strengths between 0 and 10,000 thermocycles in groups 3-5, whereas the thermocycling significantly \((p<0.05)\) decreased the bond strength in group 2. The average strengths, standard deviations, and statistical significance with the modes of failure of all specimens are summarized in Table 2. Mixed failure still occurred in all specimens even after 20,000 thermocycles in group 6. No cohesive failures were seen in any of the groups in the present study.

**Table 2. Shear bond strengths and failure modes of composite material bonded to magnetic stainless steel alloy**

<table>
<thead>
<tr>
<th>TC</th>
<th>0 cycle</th>
<th>Statistical category</th>
<th>10,000 cycles</th>
<th>Statistical category</th>
<th>20,000 cycles</th>
<th>Statistical category</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group</td>
<td>Mean ± SD (MPa)</td>
<td>Mode C / M / A</td>
<td>Mean ± SD (MPa)</td>
<td>Mode C / M / A</td>
<td>Mean ± SD (MPa)</td>
<td>Mode C / M / A</td>
</tr>
<tr>
<td>1</td>
<td>10.9 ± 6.8</td>
<td>b, c</td>
<td>3.8 ± 7.5</td>
<td>a</td>
<td>0 / 3 / 7</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>11.6 ± 2.0</td>
<td>b, c</td>
<td>10.4 ± 1.7</td>
<td>b, c</td>
<td>3 / 7 / 0</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>16.8 ± 2.0</td>
<td>b, c, d</td>
<td>14.0 ± 3.8</td>
<td>b, c, d</td>
<td>7 / 3 / 0</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>19.2 ± 4.7</td>
<td>c, d</td>
<td>19.2 ± 0.9</td>
<td>c, d</td>
<td>7 / 3 / 0</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>26.0 ± 2.0</td>
<td>d, e</td>
<td>21.1 ± 1.5</td>
<td>c, d, e</td>
<td>10 / 0 / 0</td>
<td></td>
</tr>
</tbody>
</table>

TC: Thermocycles. SD: standard deviation. Identical letters indicate that the values are not statistically different \((p>0.05)\). Mode: Failure mode \((n)\). C: Cohesive failure in the indirect composite material. A: Adhesive failure at the indirect composite material-magnetic stainless steel alloy interface. M: Mixture of cohesive failure and adhesive failure. The horizontal line for Group 1 indicates that all the cylindrical columns of the indirect composite material debonded prior to determining bond strength, with no exceptions.

**Discussion**

Because of their corrosion resistance, stainless steel alloys generally have been used for the outer components of dental magnetic assemblies and for the keepers of magnetic attachment keepers. AUM 20, which contains 19 wt% chromium, was especially designed for the yoke and keeper in the Magfit EX system according to the manufacturer’s information. Using the conventional clinical procedure, both the cast-bonding technique and the direct-bonding technique have been employed to fabricate copings with keepers. It has been reported that when a metal conditioner with a functional monomer designed for base metal alloys is applied and an adhesive acrylic resin containing a carboxylic monomer is used, great bond strength is achieved to some kinds of stainless steel.7-10 In the present study, the bond strength of one of the indirect composite materials, which can be used to veneer crowns was investigated.

It was reported that the attractive force of the Magfit system decreased when the keepers were heated during
the cast-bonding technique using silver-palladium-copper-gold alloy or gold alloy. One of the advantages of employing a resin-based composite material coping with a keeper, compared to the cast-bonding technique, is that no heat is applied to the keeper component during the fabrication procedure as well as during the direct-bonding technique. Furthermore, no especially careful surface polishing is needed to recover the attractive force.

As shown in Table 2, air abrasion and the application of a metal conditioner for base metal alloys improved the bond strength of an indirect composite material to AUM 20 steel alloy, and the application of an opaque resin also enhanced the bond strength. It was interesting that mixed failure still occurred in all the group 6 specimens even after 20,000 thermocycles, unlike the other groups. This phenomenon confirmed the effectiveness of the surface treatment in this group. Compared to groups 2 and 4, the effect of the opaque resin was remarkable, whereas it was difficult to explain the reason for the decreased bond strength in group 2 in this study. Because of the high filler proportion of 92% by weight according to the manufacturer’s information, the indirect composite material used in the present study has especially high density. Hence, this material may be difficult to bond because of the inherently small amount of the resin component. The effectiveness of the opaque resin must be chiefly attributable to the differences in the wettability of the opaque resin and the highly filled composite material to AUM 20 steel alloy as well as the viscosity of the material itself. On the other hand, it was reported that the bond strengths of the resin-modified glass ionomers were not affected by the viscosity of the materials.

One precaution to take with regard to an indirect composite resin coping is that the keeper may fracture and debond from the coping if there is not a correct amount resin around the keeper. In order to prevent such a failure, a sufficient thickness of the resin is indispensable to keep the bulk rigid.

Within the limitations of the present study, significant improvements in the bond durability of an indirect composite material bonded to a magnetic stainless steel alloy were achieved by air abrasion and the application of a metal conditioner for base metal alloys and opaque resin, followed by pre-polymerization.

Acknowledgment
The authors would like to thank Aichi Steel Corp. for supplying the AUM 20.

References

30