

## Comparative study between laser sintering and casting for retention of resin composite veneers to cobalt-chromium alloy

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The purpose of this study was to evaluate and compare the bond strengths between resin composite veneer and laser-sintered cobalt-chromium (Co-Cr) alloy with and without retention devices (Laser-R and Laser-N respectively). Cast Co-Cr alloy with and without retention devices (Cast-R and Cast-N respectively) were also prepared for fabrication technique comparison. Disk-shaped Co-Cr alloy specimens were air-abraded with alumina and veneered with a veneering system, Estenia C&B (ES) or Ceramage (CE). After 20,000 thermocycles, tensile testing was performed. Data were analyzed by ANOVA and multiple comparison test. When no retention devices were present, no significant differences were observed between Laser-N/ES and Cast-N/ES, or between Laser-N/CE and Cast-N/CE, but ES exhibited significantly higher bond strength than CE. With retention devices, Laser-R/ES, Cast-R/ES and Laser-R/CE showed no significant differences, and their retention strengths were significantly higher than that of Cast-R/CE. Compared to cast Co-Cr alloy, laser-sintered Co-Cr alloy with retention devices provided better retention durability for resin composite-veneered prostheses.

**Keywords:** Laser sintering, Co-Cr alloy, Resin, Retention strength

### INTRODUCTION

Computer-aided design and manufacturing (CAD/CAM) systems have become the mainstream method of fabricating multi-unit fixed partial denture frameworks or superstructures for dental implants<sup>1</sup>. Instead of machine milling, some CAD/CAM systems employ laser sintering as it is beneficial in creating intricate shapes, narrow cross-sections, and undercuts for retention beads. In laser sintering, a high-powered laser is used to fuse metal powders layer by layer to build the desired three-dimensional product<sup>2-9</sup>. Examples of metal powders used are titanium alloy powder or cobalt-chromium (Co-Cr) alloy powder, as used by a commercial laser sintering system EOSINT M (EOS, Munich, Germany)<sup>6</sup>.

Posterior single-unit metal-ceramic crowns fabricated by laser sintering showed a cumulative survival rate of 98.3% after 47 months<sup>7</sup>. Adaptation of Co-Cr alloy crowns fabricated by laser sintering was found to be clinically acceptable<sup>8</sup>, and Örtorp *et al.* even reported that the adaptation of three-unit Co-Cr fixed partial dentures fabricated by laser sintering was superior to conventional casting techniques<sup>9</sup>.

Adhesive bonding of veneering materials satisfies a patient's restorative needs and esthetic desires. Amongst the veneering materials, resin composites are preferred over fired porcelain for multi-unit prostheses because of their flexibility, absence of firing shrinkage, and easy handling characteristic<sup>10</sup>. However, it is difficult to prevent detachment or microleakage of resin composite veneers with adhesive bonding only. Microleakage reportedly occurred at the interface between a cast Co-Cr alloy and a resin composite veneer

because of insufficient bonding<sup>11</sup>. Therefore, instead of relying on chemical bonding alone to create the ideally strong adhesive force, macro- and/or micro-mechanical retention—such as retention beads—should be used in conjunction to maximize retention. Dental prostheses with retention devices (such as retention beads) can be formed by casting or laser sintering. Laser sintering has emerged as the superior method because it is better able to control the inter-bead distance and their undercuts *via* computer software programming.

Several studies have revealed that thermal stress induced by thermocycling weakens the adhesive bonding between resin-based materials and cast Co-Cr alloys, but priming with 10-methacryloxydecyl dihydrogen phosphate (MDP) significantly improved the bonding durability<sup>12-19</sup>. However, no information is available regarding adhesive bonding between resin composites and laser-sintered Co-Cr alloys. The purpose of this study was to evaluate the bond strength or retention strength between two resin composite veneering systems and a laser-sintered Co-Cr alloy, in comparison to those obtained with a cast Co-Cr alloy. The null hypothesis was that neither the veneering system nor the fabrication method of Co-Cr alloy would affect the retention strength between Co-Cr alloy and composite veneer if retention devices exist.

### MATERIALS AND METHODS

#### *Laser-sintered and cast Co-Cr alloy specimens*

Co-Cr alloys and resin composite veneering systems used in the present study are listed in Table 1. For fabrication by laser sintering, 48 disk-shaped Co-Cr

Table 1 Cobalt-chromium (Co-Cr) alloys, primers, and resin composites used in this study

| Name (Code)              | Manufacturer                                     | Composition  | Batch No. |
|--------------------------|--|--|-----------|
| <b>Co-Cr alloys</b>      |  |  |           |
| EOS CobaltChrome SP2     | EOS, Munich, Germany                             | Co 62–66%, Cr 24–26%, Mo 5–7%, W 4–6%,<br>Si<1.5%, Mn<1.5%, Fe<0.7%  |           |
| Cobaltan                 | Shofu Inc., Kyoto, Japan                         | Co 63%, Cr 29%, Mo 6%, other 2%  |           |
| <b>Veneering systems</b> |  |  |           |
| Estenia C&B (ES)         | Kuraray Noritake<br>Dental Inc.,<br>Tokyo, Japan | Opaque Primer: Methacrylate, MDP, solvent, others  | 00165B    |
|                          |  | Opaque OA3: Bis-GMA, methacrylate, filler (quartz,<br>composite, others), photoinitiator, pigment, others        | 00101A    |
|                          |  | Body Resin DA3: urethane methacrylate, methacrylate,<br>filler (glass, alumina), photoinitiator, pigment, others | 00067B    |
| Ceramage (CE)            | Shofu Inc.                                       | M.L. Primer: 10-MDDT, 6-MHPA, acetone  | 120942    |
|                          |  | Pre-Opaque: UDMA, aluminum silicate, 2-HEMA,<br>glass, pigment, others   | 120928    |
|                          |  | Opaque A3O: UDMA, aluminum silicate, 2-HEMA,<br>glass, pigment, others   | 110934    |
|                          |  | Body Resin A3B: UDMA, urethane diacrylate,<br>zirconium silicate, pigment, others                                | 090998    |

MDP: 10-methacryloxydecyl dihydrogen phosphate, Bis-GMA: bisphenol-A-diglycidyl methacrylate, 10-MDDT: 10-methacryloxydecyl-6,8-dithiooctanate, UDMA: urethane dimethacrylate, 6-MHPA: 6-methacryloxyhexyl phosphonoacetate, 2-HEMA: 2-hydroxyethyl methacrylate.

alloy specimens (10 mm diameter×2.5 mm thickness) were prepared from EOS CobaltChrome SP2 using EOSINT M270 laser sintering machine (EOS). Half of these specimens (24 specimens) had retention beads (Laser-R), while the remaining specimens (24 specimens) were without retention beads (Laser-N). Retention beads were approximately 230 μm in diameter, and inter-bead distance was programmed using an accompanying computer software to range between 60 and 330 μm (Fig (a)).

Forty-eight cast Co-Cr alloy specimens were fabricated from Cobaltan (Shofu Inc., Kyoto, Japan). For cast Co-Cr alloy specimens with retention beads, a double-sided tape (NICETACK, Nichiban Co. Ltd., Tokyo, Japan) of 5 mm diameter was positioned at the center of 24 disk-shaped acrylic patterns (10 mm diameter×2.5 mm thickness), and then acrylic beads of approximately 200 μm diameter (Retention Beads SS, GC Corp., Tokyo, Japan) were attached (Fig. 1(b)). Together, 24 acrylic patterns with retention beads (Cast-R) and the remaining 24 acrylic patterns without retention beads (Cast-N) were invested using a phosphate-bonded investment material (Univest Silky, Shofu Inc.), followed by casting using a vacuum induction casting machine (Argoncaster AE, Shofu Inc.) in accordance with the manufacturer's instructions.

The surfaces of Laser-N and Cast-N specimens

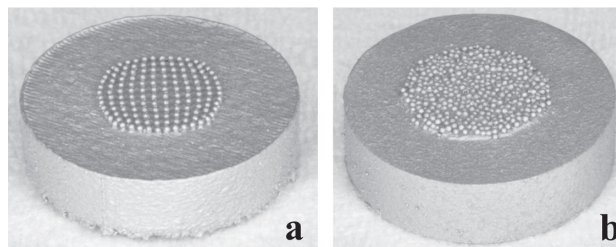


Fig. 1 Co-Cr alloy specimens with retention beads fabricated by: (a) laser sintering, and (b) casting.

were ground with 600-grit silicon carbide paper (BuehlerMet II, Buehler, Lake Bluff, IL, USA). Together, all 96 Co-Cr disk specimens were air-abraded (Jet Blast III, J. Morita Corp., Kyoto, Japan) with 50-μm alumina (Hi-Aluminas, Shofu Inc.) for 10 s at an emission pressure of 0.5 MPa with the nozzle positioned at 10 mm from the specimen surface.

#### *Bonding to resin composite veneers*

For standardization, a piece of masking tape with a circular hole (5 mm diameter) was positioned at the center of each Co-Cr alloy specimen to demarcate the

bonding area. Two resin composite veneering systems were used in this study: Estenia C&B (ES) and Ceramage (CE) (Table 1).

For ES veneering system, Estenia Opaque Primer and Estenia Body Opaque OA3 (Kuraray Noritake Dental Inc., Tokyo, Japan) were applied to the surfaces of specimens, followed by light polymerization for 180 s using a laboratory light-polymerizing unit (Alpha Light II, J. Morita Corp.). For CE veneering system, M.L. Primer and Ceramage Pre-Opaque (Shofu Inc.) were applied to the surfaces of specimens, followed by light polymerization for 90 s using the same laboratory light-polymerizing unit. Ceramage Opaque A30 (Shofu Inc.) was then applied and light-polymerized for 180 s.

An acrylic ring mold (6.0 mm diameter×2.0 mm thickness) was placed on each specimen. The ring mold was filled with the respective body resin of each veneering system, followed by light polymerization for 5 min.

#### Tensile testing

Resin composite-bonded specimens were stored in distilled water at 37°C for 24 h (baseline with 0 cycles). After 24-h water storage, half of the specimens for each combination of resin composite veneering system and Co-Cr alloy fabrication method ( $n=6$  per combination) were thermocycled 20,000 times alternately between

water baths of 4°C and 60°C, with a 1-min dwell time in each bath.

For both non-thermocycled and thermocycled specimens, cylindrical stainless steel rods were bonded to their veneer resin composites using a self-curing adhesive resin (Super-Bond C&B, Sun Medical Co. Ltd., Moriyama, Japan), as shown in Fig. 2. These bonded specimens were subjected to tensile testing in a universal testing machine (AGS-10kNG, Shimadzu Corp., Kyoto, Japan) at a crosshead speed of 1.0 mm/min. Tensile bond strengths were determined for specimens without retention beads (Laser-N and Cast-N), and retention strength per unit area was calculated for specimens with retention beads (Laser-R and Cast-R).

#### Failure mode analysis

After tensile testing, debonded specimens were observed with an optical microscope (SMZ-10, Nikon Corp., Tokyo, Japan) at 20× magnification to determine their failure modes. Failure modes were categorized as adhesive failure at resin composite-alloy interface (A), cohesive failure within resin composite (C), or complex adhesive failure at resin composite-alloy interface and cohesive failure within resin composite (A/C).

#### Statistical analysis

Tensile bond strength data of Laser-N and Cast-N specimens and retention strength data of Laser-R and Cast-R specimens were analyzed using three-way analysis of variance (ANOVA). Multiple comparisons were carried out using the Tukey-Kramer HSD test ( $\alpha=0.05$ ). Means and standard deviations of six specimens were calculated for each combination of resin composite veneering system, Co-Cr alloy fabrication method and thermocycling.

## RESULTS

#### Tensile bond strengths and failure modes of Co-Cr alloys without retention beads

Table 2 lists the mean tensile bond strengths and standard deviations of Co-Cr alloy specimens without retention beads, ranging from 1.8 MPa to 28.0 MPa. Tensile bond strengths of Laser-N/ES and Cast-N/

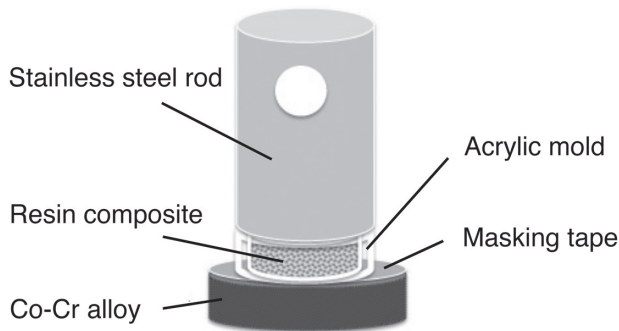


Fig. 2 Schematic illustration of bonded specimen for tensile testing.

Table 2 Tensile bond strengths and failure modes of Co-Cr alloys without retention beads

| Combination | Mean (SD) of bond strength (MPa)* |                            | Failure mode (number of specimens)** |               |
|-------------|-----------------------------------|----------------------------|--------------------------------------|---------------|
|             | 0 cycles                          | 20,000 cycles              | 0 cycles                             | 20,000 cycles |
| Laser-N/ES  | 24.0 (3.8) <sup>d, e</sup>        | 21.7 (5.0) <sup>c, d</sup> | A/C (4) C (2)                        | A/C (6)       |
| Cast-N/ES   | 28.0 (3.2) <sup>e</sup>           | 17.7 (3.2) <sup>b, c</sup> | A/C (5) C (1)                        | A/C (6)       |
| Laser-N/CE  | 12.5 (1.2) <sup>b</sup>           | 6.1 (1.3) <sup>a</sup>     | A/C (6)                              | A (2) A/C (4) |
| Cast-N/CE   | 17.0 (2.3) <sup>b, c</sup>        | 1.8 (1.5) <sup>a</sup>     | A/C (6)                              | A (4) A/C (2) |

\* Same letters indicate no significant differences ( $p>0.05$ ).

\*\* A: Adhesive failure at resin composite alloy interface; C: Cohesive failure within resin composite; A/C: Complex A and C.

ES were significantly higher than those of Laser-N/CE ( $p<0.01$ ) and Cast-N/CE ( $p<0.01$ ) before and after thermocycling. In Laser-N/ES group, no significant difference was found before and after thermocycling ( $p=0.859$ ). In contrast, tensile bond strength after 20,000 thermocycles was significantly lower in Cast-N/ES ( $p<0.0001$ ), Cast-N/CE ( $p<0.0001$ ), and Laser-N/CE ( $p=0.012$ ).

According to ANOVA results in Table 3, tensile bond strength was influenced by veneering system and thermocycling, but fabrication method of Co-Cr alloy had no significant effect. Both interactions of veneering system $\times$ thermocycling and fabrication method of Co-Cr alloy $\times$ thermocycling were significant. However, the interactions of veneering system $\times$ fabrication method of Co-Cr alloy and veneering system $\times$ fabrication method of Co-Cr alloy $\times$ thermocycling were not significant.

According to failure mode distribution shown in Table 2, 87.5% specimens exhibited complex adhesive failure and cohesive failure within the resin composite (A/C) before thermocycling, and the remaining 12.5% (two Laser-N/ES specimens and one Cast-N/ES specimen) showed complete cohesive failure (C). After 20,000 thermocycles, 75% specimens exhibited A/C failure mode and the remaining 25% (two Laser-N/CE specimens and four Cast-N/CE specimens) showed

complete adhesive failure (A). No bonding failures occurred at the interface between the resin composite and stainless steel rod.

#### *Retention strengths and failure modes of Co-Cr alloys with retention beads*

Table 4 lists the mean retention strengths and standard deviations of Co-Cr alloy specimens with retention beads, ranging from 8.0 MPa to 31.2 MPa. Before thermocycling, there were no significant differences between Laser-R/ES and Laser-R/CE ( $p=0.1505$ ), and between Cast-R/ES and Cast-R/CE ( $p=0.9461$ ). After 20,000 thermocycles, there was no significant difference between Laser-R/ES and Laser-R/CE ( $p=1.000$ ), but Cast-R/CE showed a significantly lower value than Laser-R/ES ( $p<0.0001$ ), Cast-R/ES ( $p<0.0001$ ), and Laser-R/CE ( $p<0.0001$ ).

According to ANOVA results in Table 5, retention strength was influenced by veneering system, fabrication method of Co-Cr alloy, and thermocycling. The interactions of veneering system $\times$ thermocycling and fabrication method of Co-Cr alloy $\times$ thermocycling were not significant. However, the interactions of veneering system $\times$ fabrication method of Co-Cr alloy and veneering system $\times$ fabrication method of Co-Cr alloy $\times$ thermocycling significantly affected retention

Table 3 Analysis of variance (ANOVA) results for tensile bond strengths in Table 2

| Source of variation                  | Degree of freedom | Sum of squares | Mean square | F-value | P-value |
|--------------------------------------|-------------------|----------------|-------------|---------|---------|
| A: Veneering system                  | 1                 | 2181.603       | 2181.603    | 245.338 | 0.0001  |
| B: Fabrication method of Co-Cr alloy | 1                 | 0.041          | 0.041       | 0.005   | 0.9463  |
| C: Thermocycling                     | 1                 | 889.241        | 889.241     | 100.002 | 0.0001  |
| A $\times$ B                         | 1                 | 0.030          | 0.030       | 0.003   | 0.9540  |
| A $\times$ C                         | 1                 | 59.853         | 59.853      | 6.731   | 0.0132  |
| B $\times$ C                         | 1                 | 210.841        | 210.841     | 23.711  | 0.0001  |
| A $\times$ B $\times$ C              | 1                 | 0.480          | 0.480       | 0.054   | 0.8175  |
| Residual                             | 40                | 355.690        | 8.892       | —       | —       |

Table 4 Retention strengths and failure modes of Co-Cr alloys with retention beads

| Combination | Mean (SD) of bond strength (MPa)* |                         | Failure mode (number of specimens)** |               |
|-------------|-----------------------------------|-------------------------|--------------------------------------|---------------|
|             | 0 cycles                          | 20,000 cycles           | 0 cycles                             | 20,000 cycles |
| Laser-R/ES  | 31.2 (2.5) <sup>c</sup>           | 20.9 (2.7) <sup>b</sup> | A/C (6)                              | A/C (6)       |
| Cast-R/ES   | 23.9 (0.8) <sup>b</sup>           | 21.1 (7.0) <sup>b</sup> | A/C (6)                              | A/C (6)       |
| Laser-R/CE  | 25.6 (3.4) <sup>b,c</sup>         | 21.2 (4.0) <sup>b</sup> | A/C (6)                              | A/C (6)       |
| Cast-R/CE   | 21.5 (3.5) <sup>b</sup>           | 8.0 (1.6) <sup>a</sup>  | A/C (6)                              | A/C (6)       |

\* Same letters indicate no significant differences ( $p>0.05$ ).

\*\* A: Adhesive failure at resin composite-alloy interface; C: Cohesive failure within resin composite; A/C: Complex A and C.



Table 5 Analysis of variance (ANOVA) results for retention strengths in Table 4

| Source of variation                  | Degree of freedom | Sum of squares | Mean square | F-value | P-value |
|--------------------------------------|-------------------|----------------|-------------|---------|---------|
| A: Veneering system                  | 1                 | 324.480        | 324.480     | 24.667  | 0.0001  |
| B: Fabrication method of Co-Cr alloy | 1                 | 445.301        | 445.301     | 33.852  | 0.0001  |
| C: Thermocycling                     | 1                 | 717.653        | 717.653     | 54.557  | 0.0001  |
| A×B                                  | 1                 | 76.507         | 76.507      | 5.816   | 0.0206  |
| A×C                                  | 1                 | 16.803         | 16.803      | 1.277   | 0.2651  |
| B×C                                  | 1                 | 2.167          | 2.167       | 0.165   | 0.6870  |
| A×B×C                                | 1                 | 209.167        | 209.167     | 15.901  | 0.0003  |
| Residual                             | 40                | 526.167        | 13.154      | —       | —       |

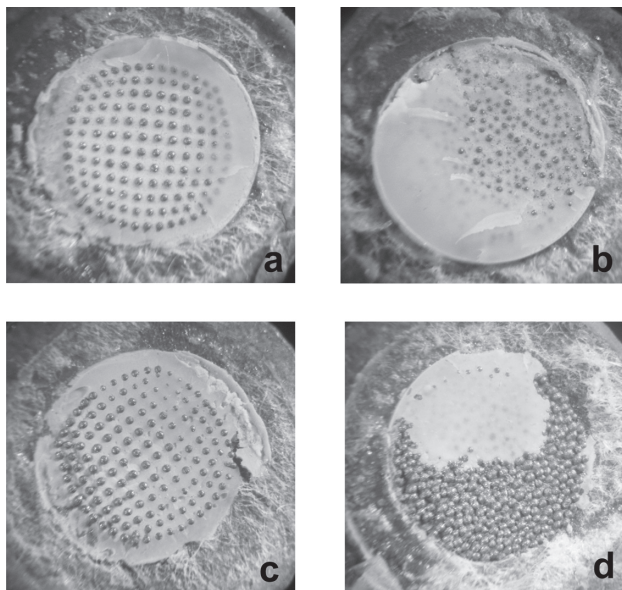


Fig. 3 Representative optical microscope images ( $\times 20$ ) of debonded surfaces after 20,000 thermocycles of: (a) Laser-R with ES; (b) Cast-R with ES; (c) Laser-R with CE; and (d) Cast-R with CE.

strength.

According to failure mode distribution shown in Table 4, all specimens exhibited complex adhesive failure and cohesive failure within the resin composite (A/C) before and after thermocycling. Figure 3 shows the representative images of failed specimens after 20,000 thermocycles. Less resin composite remained on Cast-R/CE specimen compared to Laser-R/ES, Cast-R/ES, and Laser-R/CE specimens.

## DISCUSSION

In the absence of retention devices, no significant

differences in bond strength were found between laser-sintered Co-Cr alloy and cast Co-Cr alloy. In the presence of retention devices, retention strength was affected by both the veneering system and fabrication method of Co-Cr alloy (Table 5). Therefore, the null hypothesis of this study was rejected.

Retention force generated by the retention devices depends on the undercuts and cohesive strength of the resin composite. Maximum retention strengths were reportedly achieved when inter-bead distance was one-half of or equal to the uniform diameter of the retention beads (approximately  $200\ \mu\text{m}$ )<sup>20</sup>. In this study, retention beads fabricated on laser-sintered Co-Cr alloy were located at 60–330  $\mu\text{m}$  intervals, with the diameter of the beads at 230  $\mu\text{m}$ . In the case of fabrication by laser sintering, a computer software program can be used to control the number, size, shape, and distribution of retention devices. In the present study, there were 144 retention beads within the circular bonding area.

In the assessment of different grit sizes of alumina particles (50  $\mu\text{m}$ , 110  $\mu\text{m}$ , 250  $\mu\text{m}$ , 180–330  $\mu\text{m}$ ) used for air abrasion, Barclay *et al.*<sup>21</sup> reported that grit size influenced the contact surface area of Co-Cr alloy available for bonding, but did not affect the bond strength of resin to Co-Cr alloy. These findings suggested that in Co-Cr alloy bonding, strong micromechanical retention might not be achieved with alumina particles bigger than 50  $\mu\text{m}$ . Therefore in the present study, the surfaces of laser-sintered and cast Co-Cr alloys were air-abraded using 50- $\mu\text{m}$  alumina particles.

In the absence of retention devices, results of the present study suggested that adhesive bonding of laser-sintered Co-Cr alloy is comparable to that of cast Co-Cr alloy. Between the two veneering systems, CE seemed to be more adversely affected by thermal stress than ES. In general, thermal stress derived from the difference in thermal expansion coefficients between the composite and the metal weakens adhesive bonding. Generally too, thermal stress induced by thermocycling test is a useful method for pre-clinical assessment of bonding

durability. In light of the results obtained in this study, prostheses fabricated by laser sintering and veneered with a resin composite should be clinically evaluated to verify if retention devices are still required for them.

As ES was less adversely affected by thermal stress, none of the specimens which used ES exhibited complete adhesive failure even after thermocycling. The complete or partial cohesive failure within the resin composite suggested that the adhesive force between ES and Co-Cr alloy was not lower than the cohesive strength of the resin composite. In contrast, two Laser-N/CE specimens and four Cast-N/CE specimens showed complete adhesive failure after 20,000 thermocycles. This indicated that the adhesive force at the bonding interface between CE and Co-Cr alloy was lower than the cohesive strength of the resin composite.

Differences in adhesive force at the bonding interface between ES and Co-Cr alloy and between CE and Co-Cr alloy could be attributed to the functional monomers present in the primers used. M.L. Primer of CE veneering system contained 10-methacryloxydecyl-6,8-dithiooctanate (10-MDDT) and 6-methacryloxyhexyl phosphonoacetate (6-MHPA) as functional monomers. It was reported that 6-MHPA promoted adhesive bonding to base metal alloys<sup>22</sup>. On the other hand, Estenia Opaque Primer of ES veneering system contained MDP.

Both the phosphonoacetate group of 6-MHPA and dihydrogen phosphate group of MDP may interact with and bonded to the chromium oxide in Co-Cr alloy. The methacryl groups of 6-MHPA and MDP also copolymerized with the other methacrylate monomers present in each resin composite. The chain length of alkyl group connecting the methacryl group and dihydrogen phosphate group influenced metal bonding<sup>23</sup>. In the molecular structures of MDP and 6-MHPA, the length of hydrocarbon chain segment also affected bond durability, where the longer hydrocarbon chain segment in MDP yielded more hydrophobicity and flexibility than 6-MHPA<sup>22</sup>. Therefore, differences in molecular structure between MDP and 6-MHPA may affect the tensile bond strengths produced by ES and CE.

Apart from the influence of functional monomers on adhesive bonding, the chemical components of opaque resins or mechanical properties of the resin composite may also affect bond strength or retention strength. Therefore, for dental prostheses without retention devices, further study is needed to prove that their interfacial bond strength is affected by the primer only or by both the primer and opaque resin.

## CONCLUSIONS

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

1. Without retention devices, laser-sintered Co-Cr alloy and cast Co-Cr alloy showed no significant differences in bond strength, but ES veneering system yielded higher bond strength than CE

veneering system.

2. With CE veneering system, retention devices fabricated on laser-sintered Co-Cr alloy provided better durability of retention than those of cast Co-Cr alloy.
3. With ES veneering system, retention devices on both laser-sintered and cast Co-Cr alloys showed no significant differences in retention strength.

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