

# In Vitro Tensile Bond Strength of Denture Repair Acrylic Resins to Primed Base Metal Alloys Using Two Different Processing Techniques

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#### Keywords

Removable partial denture repair; cobalt-chromium alloy; nickel-chromium alloy; autopolymerizing resin; bench cure; pressure-pot cure.

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Accepted: October 13, 2008

doi: 10.1111/j.1532-849X.2009.00499.x

## Abstract

**Purpose:** Approximately 38% of removable partial denture (RPD) failures involve fracture at the alloy/acrylic interface. Autopolymerizing resin is commonly used to repair RPDs. Poor chemical bonding of repair acrylic to base metal alloys can lead to microleakage and failure of the bond. Therefore, ideal repair techniques should provide a strong, adhesive bond. This investigation compared the tensile bond strength between cobalt-chromium (Super Cast, Pentron Laboratory Technologies, Llc., Wallingford, CT) and nickel-chromium (Rexalloy, Pentron Laboratory Technologies, Llc.) alloys and autopolymerized acrylic resin (Dentsply Repair Material, Dentsply Int, Inc, York, Pa) using three primers containing different functional monomers [UBar (UB), Sun Medical Co., Ltd., Shiga, Japan: Alloy Primer (AP) Kuraray Medical Inc., Okayama, Japan; and MR Bond (MRB) Tokyuyama Dental Corp., Tokyo, Japan] and two processing techniques (bench cure and pressure-pot cure).

**Material and Methods:** One hundred and twenty eight base metal alloy ingots were polished, air abraded, and ultrasonically cleaned. The control group was not primed. Specimens in the test groups were primed with one of the three metal primers. Autopolymerized acrylic resin material was bonded to the metal surfaces. Half the specimens were bench cured, and the other half were cured in a pressure pot. All specimens were stored in distilled water for 24 hours at 37°C. The specimens were debonded under tension at a crosshead speed of 0.05 cm/min. The forces at which the bond failed were noted. Data were analyzed using ANOVA. Fisher's PLSD post hoc test was used to determine significant differences (p < 0.05). Failure modes of each specimen were evaluated under a dissecting microscope.

**Results:** Significant differences in bond strength were observed between combinations of primers, curing methods, and alloys. Primed sandblasted specimens that were pressure-pot-cured had significantly higher bond strengths than primed sandblasted bench-cured specimens. The pressure-pot-curing method had a significant effect on bond strength of all specimens except Co-Cr alloy primed with UB. The highest bond strength was observed for both Co-Cr and Ni-Cr alloys that were sandblasted, primed with MRB, and pressure-pot cured. Co-Cr alloys primed with UB had the lowest bond strength whether bench cured or pressure-pot cured. Primed specimens generally experienced cohesive bond failures within the primer or acrylic resin. Nonprimed specimens generally experienced adhesive bond failures at the resin/metal interface.

**Conclusions:** Within the limitations of this study, MRB provided the highest bond strength to both Ni-Cr and Co-Cr alloys. Generally, bond strength improved significantly when specimens were primed. Pressure-pot curing, in most cases, resulted in higher bond strength than bench curing. The results of this in vitro study suggest that MRB metal primer can be used to increase bond strength of autopolymerized repair acrylic resin to base metal alloys. Curing autopolymerized acrylic under pressure potentially increases bond strength.

Durability of removable partial dentures (RPDs) is dependent on strong adhesion between the metal framework and acrylic resin.<sup>1</sup> Autopolymerizing resin provides for rapid, economical repairs; however, repaired units appear to lose 40% to 60% of their original transverse strength.<sup>2-4</sup> Numerous reports support the view that greater adhesion between metal and acrylic resin increases bond strength and decreases fluid microleakage.<sup>5,6</sup> The bond strength of the resin/metal interface of a prosthesis is a key factor in determining the serviceability of a restoration.<sup>6</sup>

The absence of chemical bonding between a polymethylmethacrylate (PMMA) denture base and a base metal alloy framework is a significant clinical problem with RPDs. A microscopic space exists between the metal and resin. Differences in the coefficient of thermal expansion between them may further amplify this space,<sup>7</sup> which contributes to bond failure and increased microleakage (resulting in discoloration and deterioration of the denture base material). The introduction of microorganisms that may be harbored within the crevice can also cause an unfavorable soft-tissue response.<sup>8-11</sup>

The use of appropriate adhesive primers to bond composite resins to metal has produced stable bonds and excellent esthetics by eliminating mechanical retentive features like beads, loops, pits, etc.<sup>12</sup> It also has eliminated the need for surface preparation of metal frameworks like heat treatment. Cesead opaque primer (Kuraray Co., Ltd, Okayama, Japan), containing the functional monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP), has been used to chemically bond composite resin to Co-Cr alloy. 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithione (VBATDT) improved the bond between light-cured composite resin and noble metal alloy.

Methods to bond resin to dental alloys have been used for decades. Research has yielded significant improvements in bonding materials and techniques,<sup>1,13,14</sup> such as electrolytic etching,<sup>13,15</sup> chemical etchants,<sup>14</sup> and silica coating.<sup>1</sup> Available systems like Rocatec, Silicoater MD, etc., require expensive equipment and strong, potentially corrosive chemicals. Adhesive primers are capable of chemically bonding to base metal alloys and are simple to use. NaBadalung et al<sup>16</sup> reported that an adhesive denture base resin, when bonded to nickel-chromiumberyllium dental alloys, resulted in a higher bond strength (23.9 MPa) than Rocatec-Silane-primed alloy bonded to conventional denture base resin (14.8 MPa).

The objectives of this study were to determine the tensile bond strength of autopolymerized acrylic resin to primed and nonprimed base metal alloys [nickel-chromium (Ni-Cr) and cobalt-chromium (Co-Cr)] using different curing methods. The hypotheses tested were that there should be no difference in the tensile bond strength due to alloy type, curing method, or one of the three test primers used.

## **Materials and methods**

One hundred and twenty eight cast base metal alloy ingots (Fig 1A) were provided by the manufacturer (Pentron Laboratory Technologies, LLc.). The specimens were divided into four groups of sixteen. Specimens in the control group were not primed, while those in the test groups were primed with one of three metal primers before bonding to autopolymerizing acrylic resin: UBar (UB) (Sun Medical Co, Ltd., Shiga, Japan); Alloy

Primer (AP) (Kuraray Medical Inc., Okayama, Japan); and MR Bond (MRB) (Tokuyama Dental Corp. Tokyo, Japan). Primers were applied according to the manufacturers' instructions. A 0.2 mm. circular groove was lathe cut around the vertical side of the cylindrical ingots to improve their retention in the potting resin (Fig 1A). The alloy ingots were then embedded in potting resin using a mold (Fig 1B). A small amount of boxing wax was used to secure the ingot to the mold base before pouring the potting resin (Fig 1C). After setting, the specimens were removed from the molds (Figs 1D and 2). The wax was removed, and the specimens were polished with 600 grit SiC discs, then air abraded with 50  $\mu$ m aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) for 10 seconds. They were ultrasonically cleaned in distilled water for 3 minutes, dried with an air syringe, and visually inspected to ensure complete removal of aluminum oxide particles (Fig 1E).

Autopolymerized acrylic resin (Dentsply Int. Inc., York, PA) (mixed in the ratio of 2 g of powder to 1.2 mL autopolymerized acrylic resin monomer) was fashioned into truncated cones (3 mm in diameter at the bonded surface, 5 mm in diameter at the top) using a polytetrafluoroethylene mold (Fig 1C, D). To ensure complete coverage of the bonding surface of metal, a bead and brush technique was used to place the autopolymerized acrylic resin on the primed metal surface prior to injection of the acrylic with an elastomer syringe. Figure 2 shows the assembly used to prepare the specimens.

Half the specimens were bench cured for 30 minutes; the others were pressure-pot-cured at 138 kPa for 15 minutes. All specimens were stored in distilled water for 24 hours at 37°C in a humidor (100% relative humidity).

After processing, the specimens (Fig 1E) were mounted in a loading jig and machine debonded under tension (Instron model 8501, Instron Corp., Canton, MA) at a crosshead speed of 0.05 cm/min. The forces at which the bonds failed were noted. Bond strengths were calculated in megapascals (MPa) and recorded. The failure mode of each specimen was evaluated under a dissecting microscope  $(10\times)$  and recorded.

Eight replications for each condition were tested. Means and standard deviations of bond strength were calculated. The data were analyzed using ANOVA (Statview 5.0, Cary, NC) with a factorial design. Means were compared using Fisher's PLSD post hoc test at the 0.05 significance level. Differences between means that were larger than the Fisher's PLSD (among alloys, primers, and curing methods and their interaction) were considered statistically significant (p < 0.05).

## Results

The means and standard deviations (n = 8) of bond strengths for all groups are listed in Table 1 and Figure 3. The results of the three-way ANOVA are recorded in Table 2. The main effects of alloy, cure, and primer were significant. The two-way interactions of alloy-primer and cure-primer were significant ( $p \le 0.05$ ). Two-way interaction of alloy-cure was statistically insignificant (p > 0.05). The three-way interaction of alloycure-primer was significant (p < 0.05). Fisher's PLSD for comparison of means of bond strength at the 0.05 significance level between two different alloys, between two curing methods, and among three primers were 1.4, 1.4, and 2.0 MPa, respectively.

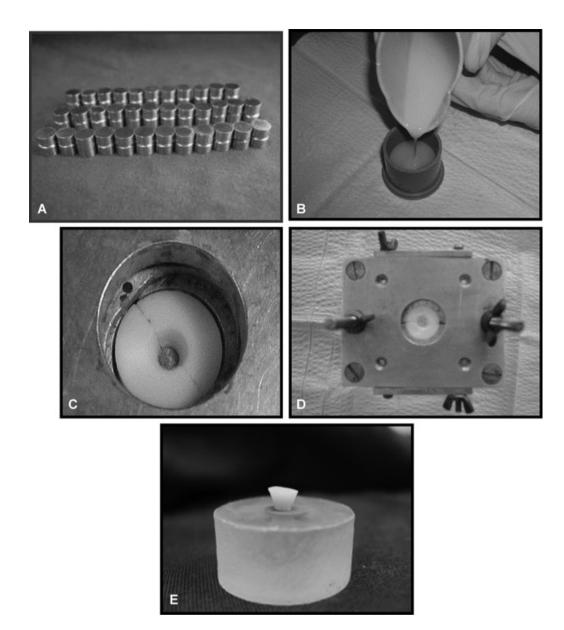


Figure 1 Specimen preparation. (A) Alloy specimens (13 mm height, 9 mm diameter). (B) Embedding metal ingots in potting resin in a mold. (C) Embedded metal alloy in the polytetrafluoroethylene mold in the jig. (D) Autopolymerizing acrylic resin on the bonding surface of the metal in the mold. (E) Bonded specimens: Autopolymerizing-acrylic-resin-treated base metal alloy.

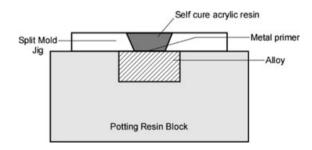


Figure 2 Assembly used to prepare bond strength specimens.

**Table 1** Means (standard deviations) of tensile bond strengths (MPa)

 between autopolymerized acrylic resin and base metal alloys

	Cobalt-C	hromium	Nickel-Chromium		
	Pressure-pot cure	Bench-cure	Pressure-pot cure	Bench-cure	
Control (No primer)	6.8 (2.3)	3.3 (1.7)	7.4 (2.1)	10.4 (3.1)	
MRB	25.6 (6.4)	11.7 (3.4)	26.9 (8.5)	14.8 (3.2)	
UB	8.6 (2.7)	8.5 (2.6)	16.8 (3.9)	11.2 (2.2)	
AP	19.2 (6.0)	14.5 (3.7)	15.3 (4.5)	11.4 (3.1)	

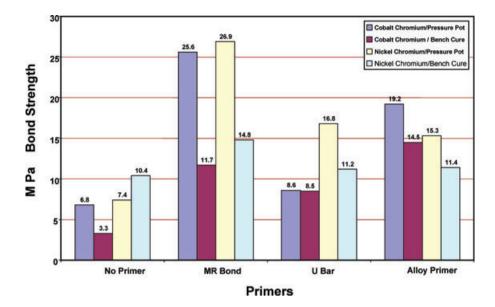


Figure 3 Mean bond strength of repair resins.

Differences between two means greater than the appropriate Fisher's PLSD intervals were statistically significant.

#### **Specimens without primer (control)**

For all nonprimed specimens, the bond strength of pressurepot-cured Co-Cr alloys had significantly higher bond strengths (6.8 MPa) than bench-cured specimens (3.3 MPa). Benchcured, unprimed Ni-Cr alloys had significantly higher bond strengths (10.4 MPa) than unprimed pressure-pot-cured specimens (7.4 MPa). When comparing unprimed, pressure-potcured Co-Cr and Ni-Cr specimens, the results were statistically similar (6.8 and 7.4 MPa, respectively). For the unprimed, bench-cured groups, Ni-Cr specimens had significantly higher bond strengths (10.4 MPa) than Co-Cr specimens (3.3 MPa).

#### **Specimens with primer**

For pressure-pot-cured Co-Cr specimens, MRB had the highest bond strength (25.6 MPa), followed by AP (19.2 MPa) and UB (8.6 MPa). UB and the nonprimed group (6.8 MPa)

	DF	Sum of squares	Mean square	F-value	<i>p</i> -value	Power
Alloy	1	128.68	128.68	7.64	0.0067	0.794
Cure	1	826.83	826.83	49.08	< 0.0001	1.000
Primer	3	2848.54	949.51	56.37	< 0.0001	1.000
Alloy * cure	1	6.78	6.78	0.4	0.5272	0.094
Alloy * primer	3	364.46	121.49	7.21	0.0002	0.987
Cure * primer	3	738.84	246.28	14.62	< 0.0001	1.000
Alloy * cure * primer	3	147.22	49.07	2.91	0.0375	0.678
Residual	112	1886.69	16.85			

had similar bond strength values. For bench-cured samples, AP had the highest bond strength (14.5 MPa), followed by MRB (11.7 MPa), UB (8.5 MPa), and nonprimed samples (3.3 MPa). For MRB and AP primed specimens, the pressure-pot-cured specimens had higher bond strengths than the bench-cured specimens; however, specimens primed with UB had statistically similar bond strengths.

MRB-primed, pressure-pot-cured Ni-Cr alloy specimens had the highest bond strength (26.9 MPa). Both AP- (15.3 MPa) and UB- (16.8 MPa) treated specimens had statistically similar (but lower) bond strength values. For pressure-pot-cured Ni-Cr specimens, all primed groups had significantly higher bond strengths than nonprimed groups (7.4 MPa). For bench-cured Ni-Cr specimens, MRB had statistically significantly higher bond strengths (14.8 MPa), when compared to UB (11.2 MPa), AP (11.4 MPa), and nonprimed specimens (10.4 MPa).

MRB and UB produced higher bond strengths with Ni-Cr in most cases, regardless of curing technique. AP produced higher bond strengths with Co-Cr than Ni-Cr in all cases.

#### **Failure mode**

The failure mode data for all groups (as recorded from observations of each specimen under the dissecting microscope at  $10 \times$  magnification) are presented in Figures 4 and 5 as mean percentage of adhesive and cohesive failure within each alloy/primer/cure group. Following Ohno et al,<sup>17</sup> adhesive failure is defined as failure along the resin/metal interface, and cohesive failure is defined as failure within the body of the resin.

All control (nonprimed) groups showed a predominantly adhesive type of failure (except for the Co-Cr pressure-pottreated specimens. They also had a significant cohesive failure component). Both Ni-Cr and Co-Cr MRB-treated groups, which were pressure-pot cured showed a high cohesive-toadhesive failure ratio. Co-Cr alloy specimens with UB showed a

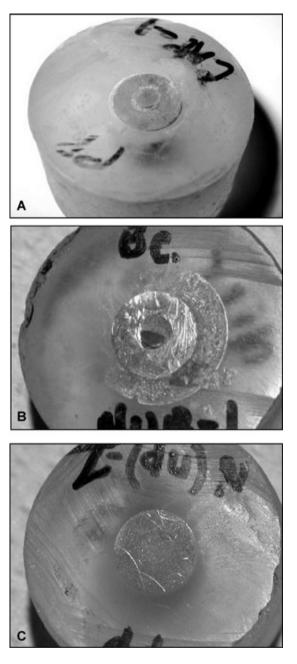


Figure 4 Failure modes for mean percentage of representative specimens. (A) 100% cohesive. (B) 40% cohesive, 60% adhesive. (C) 100% adhesive.

high adhesive-to-cohesive failure ratio regardless of the curing method. Pressure-pot-cured Co-Cr specimens with AP had a high cohesive-to-adhesive ratio. All other groups had generally similar ratios of adhesive to cohesive failure.

## Discussion

The tensile bond strength of two base metal alloys to autopolymerized acrylic resin was investigated both with and without metal primers using two curing conditions. The two base metals used were chosen because of their common use in fabrication of RPD frameworks. The three primers were tested for their ability to promote bonding of base metal alloys and autopolymerized acrylic resin.

The metal/acrylic interface of RPDs has been responsible for many restoration failures.<sup>10</sup> Strong bonding at the interface improves strength and reduces stress concentration of repaired units.<sup>2</sup> The success of a denture repair relies upon the phenomenon of adhesion, the interaction of molecules through the interface between adhesive and substance.<sup>17</sup> The strength of an adhesive joint is quantified by a specific energy of adhesion, determined by the chemical, physical, and mechanical attributes of the substrate.<sup>18</sup> This adhesive force has been dependent on the existence, strength, and amount of specific chemical bonds.<sup>17</sup>

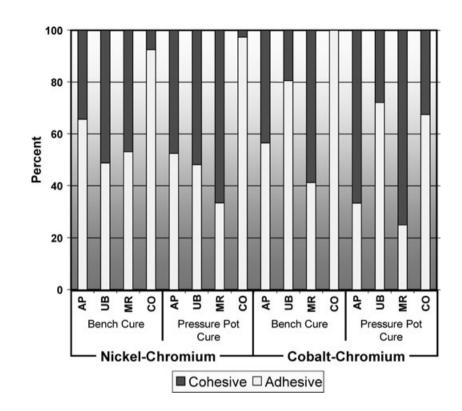
The composition and integrity of the metal surface oxide layer has been considered critical for adequate bonding. The highly polar oxide layers have inherently offered good adhesive bonding.<sup>19</sup> Some metal preconditioning has further increased bond strength, and in the case of base metal alloys, air abrasion has had the greatest effect on improving the acrylic-metal bond strength.<sup>14,20</sup> Specific surface treatments, like ultrasonic cleaning, have further enhanced bond strength.<sup>14</sup> Compared to Ni-Cr, Co-Cr alloys, which contain a higher concentration of chromium, have shown sensitivity for oxidation in lower concentrations of oxygen;<sup>14</sup> however, the Ni-Cr alloy (Rexillium V) used in this study contained between 1.6 and 1.9% beryllium. It has been reported that the addition of beryllium to some Ni-Cr alloys has controlled the surface oxidation and has resulted in more reliable bonds.<sup>21</sup>

## Without primer (control) group

Comparison of tensile bond strengths of the control (nonprimer treated) specimens showed their bond strengths to be lower than the primed specimens. Within the control group, the benchcured Ni-Cr specimens showed about 215% more tensile bond strength (3.3 vs. 10.4 MPa) than the Co-Cr specimens. The mode of failure observed for these two groups was almost entirely adhesive (Fig 5), indicating failure along the metal/resin interface. The presence of Be in the Ni-Cr alloy, as mentioned above, could have been a significant factor in promoting the formation of better surface oxidation, leading to higher bond strengths.

The bench-cured Co-Cr group had the lowest bond strength (3.3 MPa). The pressure-pot-cured specimens in this group had significantly higher bond strengths than bench-cured specimens in this same group. This supports Leong and Grant, who stated that acrylic resin repairs cured in a pressure pot or with elevated temperature resulted in increased strength compared with those carried out at room temperature and pressure.<sup>22</sup>

Bench-cured specimens of the Ni-Cr group had 40% higher tensile strengths than pressure-pot-cured specimens in the same group (10.4 MPa). This result varied from that of Leong and Grant.<sup>22</sup> One possible reason could be due to inadequate removal of minute debris left behind after polishing the alloy surface with SiC discs. The presence of trace carbon on the surface has the potential for forming carbides and creating



**Figure 5** Mean percentage of failure modes for individual alloy-primer-cure groups.

complex surface structures.<sup>18</sup> These can impact the bond strength during the adhesion process. Further research is necessary to adequately explain this phenomenon. Another possible explanation could be the type of bond formed between the metal alloy and acrylic resins. According to Bolger and Michaels,<sup>23</sup> based on the acid-base theory, the electrostatic interaction of metal oxides and polymers could result in either a hydrogen or ionic bond. Weak hydrogen bonds can easily deteriorate if exposed to water; however ionic bonds are very strong and stable and are difficult to break or weaken even in the presence of water.

The mode of tensile failure for all of the nonprimed groups was predominantly adhesive (Fig 5). This was consistent with the assumption of a lower tensile bond strength, which caused failure at the metal/resin interface. Only the nonprimed Co-Cr pressure-pot-treated group had a significant cohesive failure component. One possible reason for this could have been the pressure-pot process itself, which according to Leong and Grant<sup>22</sup> tended to promote stronger bond strength than the bench-cure technique.

## With primer group

Comparisons of tensile bond strengths in this study showed that primed specimens had higher bond strengths than nonprimed specimens. This was consistent with previous studies where primer-treated specimens were found to have significantly higher bond strengths.<sup>9,11,13,14,24-27</sup> Studies have indicated that a chemical bond was formed between the treated alloy surface and acrylic resin, which increased the tensile strength of the bond across the interface. It has been shown that the use of primers enhanced the bond strength of metal frameworks and could reduce microleakage.<sup>24,28</sup> The heat and pressure in the pressure-pot-cured method also helped the acrylic resin to polymerize better than the bench-cured technique.<sup>29</sup> Air pressure and low heat have been reported to reduce porosity in chemically accelerated materials, thus increasing repair strength.<sup>18</sup>

Comparison of the two curing processes showed that the primed, pressure-pot-cured specimens had higher bond strengths than the bench-cured specimens for both alloys. This was consistent with published results<sup>22</sup> where repairs processed in a pressure pot were stronger than those carried out at room temperature and atmospheric pressure. In previous studies, repairs completed with self-cure resin in the pressure pot exhibited transverse strengths of up to 75% of the original denturebase material.<sup>30</sup>

For both Co-Cr and Ni-Cr alloy specimens, the MRBtreated pressure-pot-cured specimens had the highest tensile bond strength (Fig 3). The tensile strength values were statistically similar for both Ni-Cr and Co-Cr alloys. The high tensile strength may have been due to increased wettability of the alloy surface using this primer.<sup>31</sup> The functional monomer in MRB is MAC-10 (11-methacryloyloxyl undecan-1, 1-dicarboxylic acid). According to the manufacturer, the methacryloyl group in MAC-10 reacts with resin, while the carboxyl group reacts with the metal alloy. It has been postulated that a strong ionic bond forms between the metal oxide on the alloy surface and the adhesive monomer (aided by the presence of surface hydroxyl groups). Similar bond mechanisms have been discussed in the literature.<sup>17,31</sup> This could possibly explain the high tensile strength between the alloys and the autopolymerizing resin in this study. The chromium oxide film formed more easily on the Co-Cr alloy than the Ni-Cr alloy.<sup>32</sup> This could also explain the high values of bond strength in Co-Cr alloys.

UBar contains the adhesive monomer 4-META, which reached the alloy surface during the brush dip technique. Monomer has been observed to adhere to the oxide film produced on the surface of alloys.<sup>33</sup> The surface treatment used in this study was sandblasting with alumina, followed by ultrasonic cleaning. This combination helped to roughen the surface of the alloys and mechanically remove debris from the surface. The ultrasonic cleaning also helped to form an oxide film derived from oxygen dissolved in water. As mentioned before, Co-Cr alloys have a higher concentration of chromium. This has created a sensitivity for oxidation in lower concentrations of oxygen.<sup>10,32</sup> The adhesive forces between 4-META and the alloy surface may be derived for several base metals.<sup>33</sup>

NaBadalung et al<sup>34</sup> reported that Meta-Dent denture base resin containing 4-META, when bonded to met-etch-treated Ni-Cr denture alloy, yielded a bond strength of 23.9 MPa. In this study, UB (4-META) primer resulted in lower bond strengths in both Co-Cr and Ni-Cr alloys. NaBadalung et al used heatpolymerized resin, whereas autopolymerizing resin was used in this study. A study by Barclay and Williams<sup>11</sup> reported a tensile bond strength of 13.16 ± 1.01 MPa when 4-META resin and silicoated Co-Cr alloys were bonded. When a meshwork was used for mechanical retention and 4-META resin was bonded to it, 6.46 ± 2.85 MPa mean tensile bond strength was reported. In this study, a bond strength of 8.6 MPa was achieved for the Co-Cr alloys without the use of any specialized surface preparations.

In both Co-Cr and Ni-Cr alloys treated with AP, the pressurepot-cured groups had significantly higher tensile strengths than the bench-cured groups. This was consistent with other studies, where higher bonding strengths have been reported when repairs done under pressure were compared with those bench cured at room temperature.<sup>22</sup> Pressure and low heat have been reported to reduce porosity seen in chemically accelerated materials, thereby increasing the repair strength.<sup>18</sup>

AP comprises VBATDT and MDP. VBATDT reacts with noble metal alloys. The phosphoric acid group of MDP chemically bonds to base metal atoms, while double bonds on the opposite end of the MDP molecule copolymerize with resin monomers.<sup>12,35</sup> The Co-Cr pressure pot specimens for the APtreated group showed statistically higher tensile strengths than those treated with UB (4-Meta). This result was consistent with those reported by Yoshida et al, where MDP primer (COP) yielded a higher shear bond strength (21.8 MPa) between photoactivated opaque composite resin and sandblasted Co-Cr alloy than specimens where a 4-META primer was used (5.5 MPa).<sup>36</sup>

Multiple studies have reported that primers help to increase the bond strengths of resin to alloys used both for fixed and removable partial dentures.<sup>10,37</sup> In this investigation of three primers, MRB (containing MAC-10) showed significant enhancement of bond strengths of autopolymerizing resin cured under pressure to primer-treated base metal alloys. Most failures were cohesive, indicative of the low tensile strength of autopolymerizing resin. Trends in this study indicated that the higher the bond strength, the higher the percentage of cohesive failure within the autopolymerizing acrylic resin, consistent with results reported in other studies.<sup>3</sup>

# Conclusions

Within the limitations of this study, the following can be concluded:

- 1. All except one of the primed groups provided higher tensile bond strength than the nonprimed groups.
- 2. Among the primed groups, Co-Cr and Ni-Cr alloys primed with MRB and pressure-pot cured produced the highest tensile bond strengths.
- 3. Among the bench-cured specimens, Ni-Cr alloys primed with MRB resulted in the highest tensile bond strength.
- 4. In most cases, pressure-pot curing resulted in higher tensile bond strength than bench curing.

Clinical significance: A simple, effective, economical, and durable method of RPD repair has provided clinically relevant results. The results of this in vitro study suggest that metal primers with Co-Cr and Ni-Cr alloys can be used to increase tensile bond strength of the repairs and enhance longevity of RPDs. Curing the repaired surface under pressure further increased the strength of the repair.

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