

RESEARCH AND EDUCATION

Bond strength of ceramics heat-pressed onto three dental alloys



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Metal-ceramic restorations continue to be common in fixed prosthodontics because of their high mechanical strength and predictability.^{1,2} The conventional porcelain layering (CPL) technique has been used in dental laboratories for many decades. However, substantial variation exists in the mechanical properties of different alloys in association with various veneering porcelains, depending on the clinical situation and the experience of the dental laboratory technician. The heat-pressing technique with heat-pressed ceramics is a popular method for fabricating ceramic restorations.^{3,4} It was developed to optimize working procedures and to increase the productivity and efficiency of dental laboratories. The press-on-metal (PoM) technique is an innovative method for processing metal-ceramic restorations.⁵ The PoM technique provides for fully anatomic pressing and consistent quality. It also exhibits superior characteristics in terms of esthetics, marginal fit, and intaglio accuracy.⁶⁻⁸ To improve the metal-ceramic chemical bond, indium and tin are

ABSTRACT

Statement of problem. The press-on-metal (PoM) technique has been used as an alternative fabrication method for metal-ceramic restorations. However, how the PoM technique compares with the conventional porcelain layering (CPL) technique under a variety of conditions is unclear.

Purpose. The purpose of this in vitro study was to compare the bond strength of 3 alloy substrates with heat-pressed ceramics or conventionally layered porcelain before and after thermocycling.

Material and methods. Specimens (n=5) of Au, Pd, and Ni-Cr alloys were veneered with heat-pressed ceramics or conventionally layered porcelain. The 3-point bend test was conducted according to the International Organization for Standardization standard 9693-1 as bond strength before and after thermocycling. The metal-ceramic interfaces were characterized by field emission scanning electron microscopy (FESEM) and energy dispersive X-ray spectroscopy (EDS). Two- and 3-way ANOVA followed by the Tukey honestly significant difference (HSD) test were used to analyze the data ($\alpha=.05$).

Results. Significantly lower mean bond strength was recorded for the Au and Pd alloys of the PoM group than for those of the CPL group ($P<.05$). CPL-Au demonstrated the highest bond strength of 50.2 ± 2.0 MPa, whereas PoM-Pd showed the lowest bond strength of 31.8 ± 2.7 MPa; significant differences were found among all groups ($P<.05$). After 20 000 thermocycles, CPL-Au showed significantly reduced bond strength value ($P<.05$). A value of approximately 40 MPa was observed in all groups except for PoM-Pd (26.5 ± 1.6 MPa, $P<.05$). The metal-ceramic interface resulting from the PoM technique revealed 2- to 20- μ m pores, with more defects observed in the PoM-Pd group than in any of the other group.

Conclusions. Defects and an oxide layer were formed at the metal-ceramic interface during the heat-pressing process, especially for the Pd alloy. After thermocycling, PoM-Pd had the lowest bond strength value, although it exceeded the minimum 25 MPa of the ISO 9693-1 standard. The Au and Ni-Cr alloys exhibited similar levels of porcelain bond strength with both techniques. (J Prosthet Dent 2019;121:867.e1-e5)

added to noble metal alloys to increase oxidization, and beryllium may be added to base metal alloys.^{9,10} Furthermore, oxidation treatment before porcelain application is important for increasing the metal-ceramic bond strength.¹¹ The compositions of the opaque porcelain and

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Clinical Implication

The press-on-metal technique is an acceptable alternative fabrication method for metal-ceramic restorations, especially when metal substrates are fabricated from Au or Ni-Cr alloys.

the firing process have a significant effect on metal-ceramic adhesion.¹²

Thermal changes affect the service life of metal-ceramic restorations.^{13,14} The thermocycling test, designed to replicate clinical conditions, produces changes in material volume because repeated temperature alternations induce mechanical stresses and direct crack propagation through the bonded interfaces.¹³ The interaction of the thermal and mechanical stresses in the aqueous solution directly affects the metal-ceramic bond.^{15,16} Sebastiani et al¹⁷ suggested that improved adhesion is possible by controlling interfacial stress and defect distribution. Solá-Ruiz et al⁵ reported that crowns produced by the conventional technique resist fracture better than those produced by the PoM technique. However, Henriques et al¹⁸ reported contradictory results, and Schweitzer et al¹⁹ indicated no significant difference in bond strength between pressed ceramics and layering porcelain. However, the authors are unaware of a study evaluating the bond strength of PoM after thermocycling and the interactions among alloy types, fabrication methods, and thermal changes to elucidate the bonding mechanism.

Numerous methods, including shear tests, tensile tests, a combination of shear and tensile tests, bend tests, and torsion tests, have been designed to evaluate metal-ceramic bond strength.²⁰ Among them, the 3-point bend test is used to evaluate flexural strength in a nonductile solid, such as porcelain. The porcelain is fired on the tensile face of a metal strip and tested by bending, with the results presented as metal-ceramic bond strength.^{11,21-24} The International Organization for Standardization (ISO) standard 9693-1:2012 recommends that the minimum acceptable bond strength is 25 MPa.²¹

This *in vitro* study evaluated the effects of thermocycling on porcelain bond strength which were compared between the PoM and CPL techniques using 3 representative alloys. The null hypothesis was that the 3 alloy substrates used with both techniques after thermocycling would exhibit similar levels of porcelain bond strength.

MATERIAL AND METHODS

The metal substrates were fabricated from Au alloy (Lodestar; Ivoclar Vivadent AG), Pd alloy (Spartan Plus; Ivoclar Vivadent AG), and Ni-Cr alloy (Unitbond; Jensen). The main chemical compositions of these alloys

Table 1. Main chemical compositions (wt %) of alloys (manufacturers' values)

Alloy	Brand	Au	Pd	Cu	In	Ga	Ni	Cr	Mo	Al	Other
Au	Lodestar	51.5	38.5	-	8.5	1.5	-	-	-	-	Ru<1.0, Re<1.0
Pd	Spartan	2.0	78.8	10.0	-	9.0	-	-	-	-	Ir<1.0, Li<1.0, Ge<1.0
Ni-Cr	Unitbond	-	-	-	-	-	78.5	12.5	5	2	Be, Ti

provided by the manufacturers are listed in Table 1. Two veneering porcelains—leucite-based ceramics (IPS InLine PoM; Ivoclar Vivadent AG), tested for PoM, and a feldspar-based ceramic (VMK Master; Vita Zahnfabrik), for the control CPL—were used. The metal-ceramic specimens met the dimensional requirements of ISO standard 9693-1:2012 (n=5). All treatments of the alloys and veneering porcelains were carried out according to the manufacturers' recommendations. Rectangular resin patterns (Light Curing Trayplates; Vertex) were invested with a phosphate-bonded investment material (Adenta-Vest CB; Adentatec) and cast in an argon atmosphere by using a centrifugal casting apparatus (Argoncaster-C; Shofu). The metal specimens (25×3×0.5 mm) were obtained after polishing with 400- and 1000-grit abrasive paper. All specimens were abraded with 110- μ m Al₂O₃ particles (Korox; Bego) for 15 seconds at an operating distance of 10 mm and a pressure of 0.3 MPa. The surface morphology was then examined by using a field emission scanning electron microscope (JSM-7401F; JEOL). For the process of veneering porcelains, a 0.2-mm-thick opaque porcelain layer was fired twice on the center section of the specimens by applying the respective opaque porcelains. For the PoM group, the wax patterns were fabricated on the opaque layer. Then, the corresponding procedures were executed, and the restorations were pressed in a furnace (EP 600; Ivoclar Vivadent AG). The dentin porcelain was fired twice in a ceramic furnace (Vacumat 40; Vita Zahnfabrik) for the CPL group. Ceramic dimensions of 8×3×1 mm were obtained, and glaze porcelain was applied and fired.

The metal-ceramic specimens were immersed in deionized water for 20 000 thermocycles at between 5°C and 55°C with a dwell time of 30 seconds by using a thermocycling device (TBN-971105; Ten Billion Technology). The 3-point bend test was conducted by using a universal testing machine (AG-1000E; Shimadzu) at a crosshead speed of 0.5 mm/min. The bond strength was calculated using the following formula:

$$\sigma = \frac{3PL}{2bd^2}$$

where σ =bond strength; P=load at fracture; L=support span; b=width of the specimen; and d=thickness of the specimen.

Two-way ANOVA, 3-way ANOVA, and the Tukey honestly significant difference (HSD) tests were used to

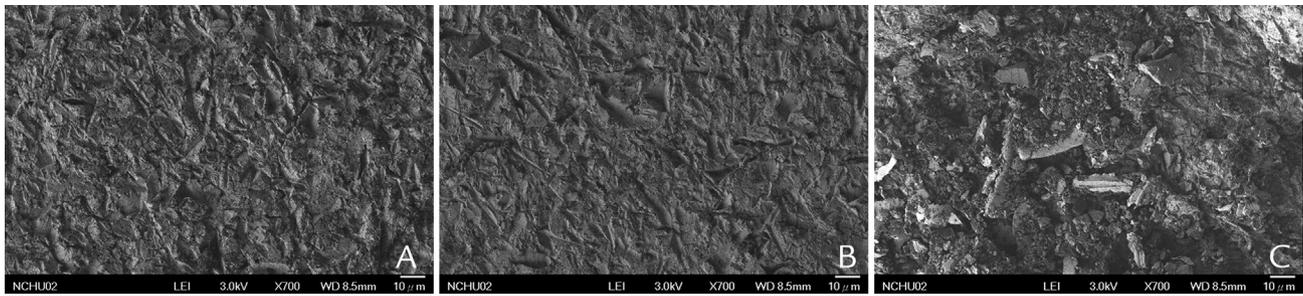


Figure 1. Scanning electron microscope images of airborne-particle-abraded alloy surface morphology (original magnification $\times 700$). A, Au alloy. B, Pd alloy. C, Ni-Cr alloy.

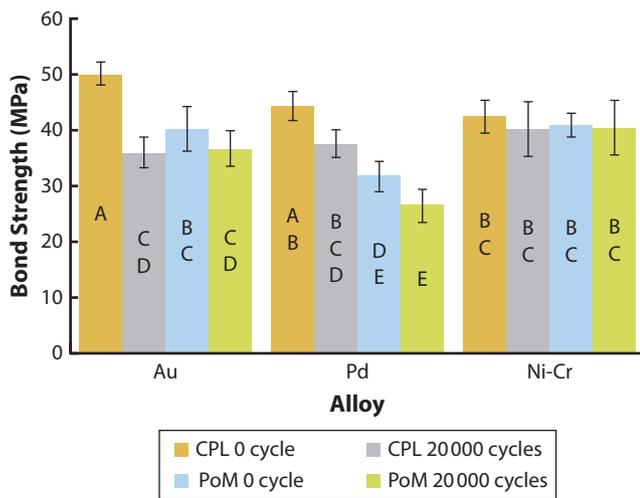


Figure 2. Mean \pm standard deviation bond strength of specimens before and after thermocycling. Different uppercase letters denote statistical significance ($P < .05$).

determine significant differences among alloy types, fabrication methods, and thermocycling in terms of their effects on bond strength by using a statistical software program (JMP 14; SAS Institute) ($\alpha = .05$).

Metal-ceramic interface morphology was visually examined by field emission scanning electron microscopy (FESEM). After the 3-point bend test, elemental composition analysis was conducted on the residual ceramics of the alloy surface, and all fractured specimens were examined to determine the failure mode. The failure modes were classified as adhesive failure occurring along the interfacial region between the opaque porcelain and metal, cohesive failure in the veneering porcelain, or mixed failure which included both modes.¹⁶

RESULTS

The SEM images of the metal substrates after airborne-particle abrasion are shown in Figure 1. The Ni-Cr alloy showed clear differences in surface morphology when compared with the other alloys, with spherical upheaval of 30 to 100 μm in diameter. The summarized results of

Table 2. Two-way ANOVA results of relative bond strength

Source	Sum of Squares	df	Mean Square	F	P
Alloy	238.1	2	119.0	14.354	<.001
Method	503.2	1	503.2	60.675	<.001
Alloy \times method	173.9	2	86.9	10.484	<.001
Error	199.0	24	8.3		
Corrected total	1114.1	29	38.4		<.001

Table 3. Three-way ANOVA results of relative bond strength

Source	Sum of Squares	Df	Mean Square	F	P
Alloy	496.4	2	248.2	26.098	<.001
Method	572.4	1	572.4	60.183	<.001
Alloy \times method	280.2	2	140.1	14.729	<.001
Thermocycling	365.0	1	365.0	38.378	<.001
Alloy \times thermocycling	155.8	2	77.9	8.188	<.001
Method \times thermocycling	60.8	1	60.8	6.392	.015
Alloy \times method \times thermocycling	72.9	2	36.5	3.837	.029
Error	456.5	48	9.5		
Corrected total	2460.0	59	41.7		<.001

the bond strength before and after thermocycling are shown in Figure 2. Before thermocycling, the bond strength values of the Au and Ni-Cr alloys of PoM were lower than those of CPL. CPL-Au (50.2 ± 2.0 MPa) and PoM-Pd (31.8 ± 2.7 MPa) showed the highest and lowest mean values, and these values differed significantly from those of the other groups ($P < .05$). From the 2-way ANOVA (Table 2), the alloy types, fabrication methods, and their interactions had a significant effect on the bond strength ($P < .001$). The mean value of all groups decreased after thermocycling, especially that of CPL-Au, which was significantly reduced ($P < .05$). A value of approximately 40 MPa was observed in all groups except for PoM-Pd (26.5 ± 1.6 MPa, $P < .05$). For the PoM group, the bond strength was negatively affected by thermocycling, but the Ni-Cr alloy expressed a high bond strength value of 40 MPa that was not affected by the fabrication method or thermocycling. The results of the post hoc tests showed statistically significant differences among the alloy types, fabrication methods, and thermocycling

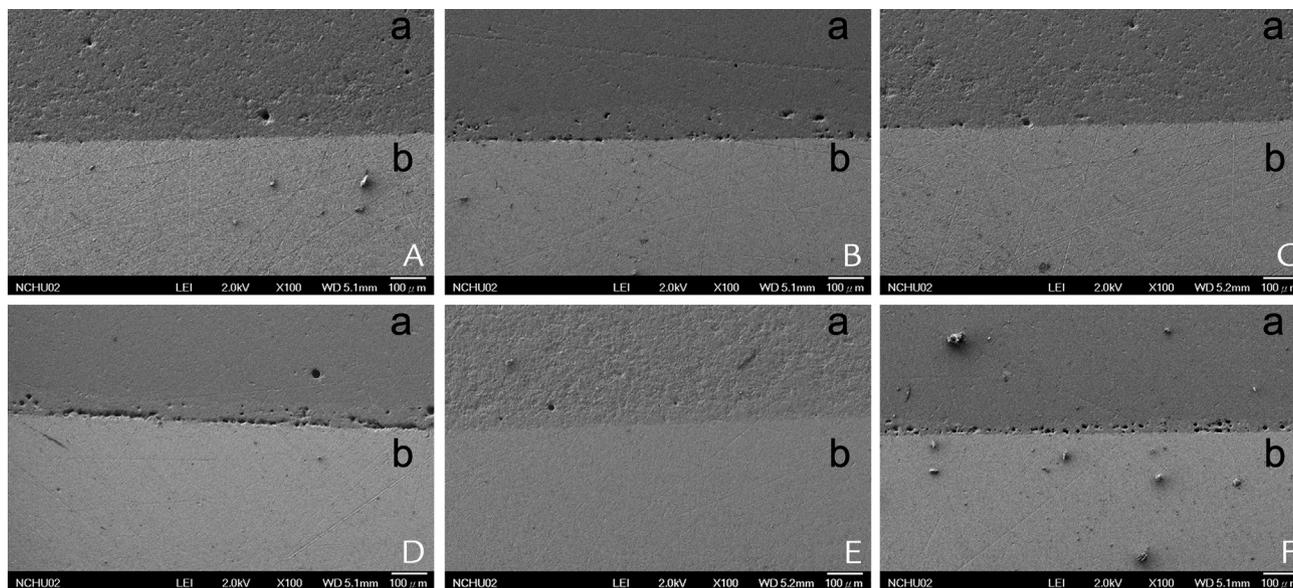


Figure 3. Interface of metal-ceramic specimens (original magnification $\times 100$). A, CPL-Au alloy; B, PoM-Au alloy; C, CPL-Pd alloy; D, PoM-Pd alloy; E, CPL-Ni-Cr alloy; F, PoM-Ni-Cr alloy. (a) Metal substrate; (b) ceramic. CPL, conventional porcelain layering; PoM, press-on-metal.

in terms of their effects on bond strength ($P=.029$) (Table 3).

The interface of metal-ceramic specimens is shown in Figure 3. Few pores were found at the metal-ceramic interface of CPL-Pd specimens. The PoM group revealed more significant pores, which varied between 2 and 20 μm in size. Moreover, the PoM-Pd group showed more pores and structural defects than any other group. After thermocycling and the bend test, the surfaces of the specimens showed different features. All CPL-Au and 2 of the 5 PoM-Au specimens demonstrated intact opaque layers, with cohesive fracture in the veneering porcelain. No residual porcelain, not even the oxide layer, was observed in the center section of any of the Pd and in 3 of the 5 PoM-Ni-Cr specimens, pointing to the delamination of the oxide layer and metal.

The analyzed compositions of the residual porcelain on the metal substrates after the bend test are presented in Table 4. In both groups, O, Si, K, Al, Na, and Ca were detected. Trace amounts of Ti, Sn, and Ba were observed in the CPL group, and a higher proportion (27.4%) of Zr was detected in the PoM group.

DISCUSSION

The Au and Ni-Cr alloy substrates exhibited similar bond strength with both techniques after thermocycling, but the Pd alloy substrates exhibited significantly lower bond strength with the PoM technique. Therefore, the null hypothesis was rejected.

In the present study, CPL-Au specimens showed significantly higher bond strength. This was attributed to the In content of the Au alloy and the Sn content of the

Table 4. Compositions of opaque porcelain attached to metal substrates after bend test (wt %)

Group	O	Si	Ba	K	Al	Zr	Ti	Na	Sn	Ca
CPL	51.0	16.9	5.6	5.6	5.0	4.9	4.9	4.5	1.1	0.6
PoM	44.9	14.7	-	5.0	3.9	27.4	-	3.0	-	1.1

ceramics. A metal-ceramic interface possessing an intermediate layer with elemental interpenetration between the oxide layer and the ceramic improves the adhesive bond.²² After airborne-particle abrasion, an area of roughness and upheaval of between 30 and 100 μm in diameter was produced on the surface of the Ni-Cr alloy, which was beneficial to the adhesive bond.¹⁸ The PoM group showed lower bond strength than the CPL group, and its metal-ceramic interfaces revealed numerous structural defects. This poor performance might have been because of gases emitted from the metal surface when the wax pattern was eliminated. According to the observations of fractured specimens, the complete oxide layer fractured in the Pd and Ni-Cr specimens. As a result, the heat treatment in air during the lost-wax process forms more oxide layer, which weakens the metal-ceramic bond. Metal-ceramic adhesion also decreases when porosity increases. Molten alloys with high Pd content reabsorb and react easily with atmospheric gas and then further condense and release during the solidification process to form pores and defects identified in the Pd specimens. Khmaj et al²³ reported that Au alloy specimens with CPL had higher bond strengths than with PoM, and Pd alloy with PoM had the lowest bond strength, consistent with the results of the present study.

Metal-ceramic bond strength is an important factor in the service life of dental metal-ceramic restorations. In the present study, all groups had a bond strength value of approximately 40 MPa after thermocycling, except for PoM-Pd, although it exceeded the 25 MPa specified in the ISO standard. The characteristic variation of the metal-ceramic interface during the heat-pressing process is a limitation of this study. Further studies are needed to explain this behavior and to improve the parameters of the heat-pressing procedure and processing devices. Long-term experiments are recommended for evaluating clinical applications.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. The ceramics heat-pressed onto the 3 alloys attach well and exhibit acceptable metal-ceramic bond strength, even after 20 000 thermocycles.
2. The formation of excess pores and an oxide layer at the metal-ceramic interface during the heat-pressing process and lower bond strength were observed in the PoM-Pd group.
3. Au and Ni-Cr alloys are recommended as metal substrates with heat-pressed ceramics. However, the long-term performance of the Pd alloy is of concern.

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