Comparison of Bond Strength of a Pressed Ceramic Fused to Metal versus Feldspathic Porcelain Fused to Metal

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Purpose: To compare the debonding/crack initiation strength (D/CIS) of a low-fusing pressable leucite-based glass ceramic (PC) fused to metal to a feldspathic porcelain (FP) fused to metal.

Materials & Methods: As per ISO 9693:1999, 40 rectangular metal specimens (25.0 mm × 0.5 mm × 3.0 mm) were prepared. Twenty of the specimens were cast in a base metal nickel-chromium alloy (BA), and 20 were cast in a noble metal palladium-silver alloy (NA). Ten randomly selected NA and BA alloy specimens had FP applied. The remaining 10 NA and BA alloy specimens had ash-free wax patterns applied, the metal-wax complexes invested, and were pressed with a PC. The dimensions of the ceramic specimens were 8.0 mm × 1.0 mm × 3.0 mm, creating a combined metal-ceramic complex thickness of 1.5 mm. All specimens were subject to a three-point bending test at a crosshead speed of 1.5 mm/min. Fracture loads were recorded in Newtons and D/CISs calculated by the formula $\tau_b = k \times F_{fail}$.

Results: Mean D/CISs, measured in MPa (standard deviations): NA-FP 32.56 (4.62), NA-PC 30.23 (5.06), BA-FP 30.98 (4.41), and BA-PC 31.81 (3.48). A two-way ANOVA ($p > 0.05$) did not demonstrate significant difference between groups.

Conclusion: The debonding/crack initiation strength of a low-fusing pressable leucite-based glass ceramic fused to metal was equivalent to that of a feldspathic porcelain fused to metal.

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INDEX WORDS: Schwick crack-initiation test, three-point bending test, pressable ceramic, metal-ceramic bond strength

CONVENTIONAL PORCELAIN fused to metal (PFM) restorations have demonstrated superior fracture resistance over all-ceramic restorations because of an alloy framework.[1] According to Craig et al, the failure rate of most PFM restorations at 10 years in vivo has been found to be significantly less than all-ceramic restorations;[2] however, ceramics used for all-ceramic restorations have many advantages over those traditionally used for PFM restorations. All-ceramic materials have higher flexural and compressive strength with less porosities than conventional feldspathic porcelain (FP), provide better esthetics with increased translucency, shrink less during processing, and have excellent marginal fit.[3-6] Moreover, pressed ceramics (PC), used for all-ceramic restorations, have the additional advantage of being technically less challenging by use of the lost-wax technique. This allows for the convenience of a full-contour ceramic wax-up as opposed to the more technique-sensitive layering method. Conceptually, combining such a ceramic with the clinically proven reinforcing ability of a metal framework would be advantageous; however, PCs

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could not be used with a metal framework because of mismatches in the ceramic fusion vs. alloy casting temperatures and differences in the coefficient of thermal expansion (CTE) of the two substrates.[7-9]

The metal-ceramic (MC) bond interface is critical in the functional and esthetic success of dental MC prostheses. Four factors contribute to the strength of the MC bond:

1. *Chemical bond*: dictated by the oxide layer formed on the metal substrate that forms metallic, ionic, and covalent bonds with oxides in the ceramic opaque.
2. *Mechanical interlocking*: the ceramic physically engaging undercuts on the metal substrate surface.
3. *Van der Waal's forces*: attraction based on molecular charge.
4. *Compressive forces*: those based on CTE.[7-10]

The chemical bond and mechanical interlocking are believed to play the most prominent role in the bond strength of ceramic to metal.[7-10] Van der Waal's forces are a minor contributor to MC bond strength.[9] Compressive forces depend on, for example, the geometric design of a PFM coping that can draw the veneering ceramic toward the metal upon cooling after reaching fusion temperature.[9]

Significant variables that have affected bond strength at the MC interface have been studied. Drummond et al[11] demonstrated that the bond strengths of porcelain fused to noble alloys (NA) were significantly higher than the bond strengths of porcelain fused to predominately base metal alloys (BA). Hammad et al[12] found that the firing temperature of ceramic opaque could have an effect on bond strength. The investigators demonstrated that the firing of opaque porcelain 65°F above the manufacturer's recommendation significantly increased the MC bond interface strength. Alternatively, thermocycling and wet storage used to simulate oral conditions over several years have shown an ability to significantly weaken MC bond strengths.[13] Thermocycling is destructive via the induction of crack propagation caused by disruption of material matrices from sudden changes in temperature.[14,15] Furthermore, thermocycling can change the marginal gap dimensions of a dental restoration creating a fluctuation in gap volume that can pump pathogenic bacteria in and out of the tooth-restoration interface.[14]

With MC restorations, the casting temperature of the alloy must be a minimum of 170 to 280°C above the fusing temperature of the ceramic to avoid distortion in metal frameworks.[9] NAs (containing at least 25% total gold, palladium, and platinum) used for dental prostheses have a casting temperature of 1150 to 1350°C.[16] BAs used for dental prostheses have a casting temperature of 1300 to 1860°C.[16] Traditional leucite-based PCs have a fusion temperature of 1150 to 1200°C, making them incompatible with most contemporary alloys.[4,6]

The CTE dictates the rate at which a material contracts during cooling. This contraction rate can create strong shear stresses at an MC interface if the two substrates are mismatched, leading to failure via ceramic crack propagation.[7,8] Dental ceramics are brittle materials that are stronger when in compression and more resistant to applied tensile forces. Therefore, it is beneficial to have some residual shear stress after cooling, via a small CTE mismatch, to take advantage of this physical property. According to Wataha, the CTE of metal should be slightly higher (approximately 0.5×10^{-6}/°C) than that of the applied ceramic, putting the ceramic in residual compression upon cooling.[17] Most PFM alloys have a CTE of 13.5 to 14.5×10^{-6}/°C.[8] Traditional leucite-based PCs have a CTE of 14.5 to 18.0×10^{-6}/°C,[4] and lithium disilicate-based PCs have a CTE of 10.0 to 11.0×10^{-6}/°C,[18] creating a thermal incompatibility with most PFM alloys.

Recently, the development of a low-fusing pressable leucite-based glass ceramic, compatible with PFM alloys in both processing temperature and CTE, has allowed for its merging with the traditional strength of an alloy framework.

Various authors have investigated the experimental methods for testing MC bond strength. Della Bona and Van Noort[19] criticized the planar interface test for being governed by the cohesive strength of the ceramic material used rather than the adhesive bond at the MC interface. Via finite elemental analysis, the authors suggested that this was due to the non-uniform stress distribution generated in the planar shear test design. Alternatively, they contended that in tensile bond strength tests, fracture always occurred at or near the MC interface (adhesive failure). Hammad and Talic[20] reviewed the literature of bond tests including shear tests (pull or push through, planar, etc.) and tensile tests (flexure and torsion), and concluded that the available tests only approximated the bond strength of the MC system, and that absolute strength remained elusive. The authors felt that true bond strength could only be achieved if the cohesive strength of the FP exceeded that of the MC bond, and the CTE of the metal and ceramic were identical. They suggested that the most reliable type of bond test should have minimal experimental variables, and that testing should be standardized. Papazoglou and Brantley[21] found that different test geometries created stress patterns resulting in incomparable bond strength values. They concluded that residual interfacial stresses resulting in background stress would also need to be calculated for accurate bond assessment. They felt that this can only be done by the exact matching of the CTE of the metal and ceramic, and is an impossibility since the ceramic and metal thermal contraction coefficient do not coincide during cooling.

Due to the abundance of various testing methodologies, which has limited the ability of investigators to compare the results of different MC bond strength studies, the International Organization for Standardization (ISO) standardized MC bond testing through the Schwickerath crack-initiation test, a three-point bending test (ISO/FDIS 9693: 1999).[22]

The purpose of this study was to compare the debonding/crack-initiation strength of a low-fusing pressable leucite-based glass ceramic fused to metal to a feldspathic porcelain fused to metal, utilizing the ISO standard Schwickerath crack-initiation test.

Materials and Methods

Forty rectangular metal specimens were cast: 20 palladium-silver-based NA (Argelite 60, Argen, San Diego, CA) and 20 nickel-chromium-based BA (Ugirex III, Ugindentaire, France) (Table 1). To fabricate the metal specimens, casting wax strips with dimensions of approximately 26.0 mm × 0.6 mm × 3.5 mm were cut using a rectangular cutting instrument from sheets of 22-guage wax (Part #32245, Kerr/Sybron, Romulus, MI). Each wax specimen was then connected to a wax sprue and invested in a phosphate-bonded investment (Finesse, Ceramco, Burlington, NJ) mixed under vacuum for 1 minute. After a 1-hour setting time, the specimens were inserted in a room temperature burnout oven (Accu-Therm II 250, Heraeus Kulzer [Jelenko], South Bend, IN) and gradually heated to 800°C. All specimens were cast (NA at 1370°C and BA at 1400°C) in a centrifugal casting machine (74 Exac-U-Cast, Handler, Westfield, NJ), divested and then sandblasted (Micro-etcher, Model ERC, Danville Engineering Inc., Danville, CA) for 15 seconds with 50 μm aluminum oxide at 60 psi to remove residual investment material. Specimens were then ultrasonically cleaned for 30 minutes in a distilled water bath. All specimens were degassed before processing to prevent hydrogen absorbed during the casting process from inducing porosities in the applied porcelain, and to create an oxide layer to promote a strong chemical MC bond.[10] The metal specimens were hand shaped to a dimension of 25.0 mm in length and 3.0 mm in width using a polisher (Phoenix Beta, Buehler, Lake Bluff, IL). Then they were

Table 1. Metals Used

Metal	*Composition (%)*	*Elastic Modulus (MPa)*	*Vicker's Hardness (HV/10)*	*Tensile Str. (MPa)*	*Yield Str. (MPa)*	*Elongation (%)*	*Melting Range (°C)*	*Casting Temp. (°C)*	*CTE ($\times 10^{-6}$/°C)*
Argelite 60 (Noble) Argen, SD, CA	Pd 59.9 Ag 28.0 In 5.0 Sn 5.0 Zn 2.0 Ru 0.1	137.00	250	640 to 697	460 to 540	20 to 25	1230 to 1305	1370	14.5 to 14.6
Ugirex III (Base) Ugindentaire, France	Ni 60.0 Cr 26.0 Mo 9.4 Si 2.4	200.00	250	550 to 650	340 to 450	4 to 8	1150 to 1280	1400	14.3 to 14.9

Table 2. Ceramics Used

Ceramic	*Fusion Temperature (°C)*	*Flexural Stress (MPa)*	*CTE ($\times 10^{-6}$/°C)*	*Solubility (μg/cm^2)*
CPC-MK (Pressed) Chemichl AG, Liechtenstein	920	114	13 ± 0.5	20
Ceramco II (Feldspathic) Ceramco, Burlington, NJ	940	70	12.5 ± 0.5	17

attached to the polisher wheel with cyanoacrylate and shaped to a thickness of 0.5 mm. The samples were subsequently removed and cleaned with acetone. All metal samples were measured with a digital caliper (Series #500, Mitutoyo, Japan), sandblasted for 15 seconds with 50 μm aluminum oxide at 60 psi, and ultrasonically cleaned for 30 minutes in a distilled water bath before ceramic application.

Ten randomly selected NA and BA metal specimens had FP applied via the use of a custom Teflon mold (Dupont, Wilmington, DE) to dimensions of approximately 8.0 mm × 1.0 mm × 3.0 mm as follows: two layers of opaque (Ceramco II, Ceramco, Burlington, NJ) were applied to the experimental surface and fired individually, under vacuum, in a calibrated porcelain oven (Commodore 100 UFF, Heraeus Kulzer [Jelenko]) to 990°C. Then, body porcelain (Ceramco II) was vibrated and condensed onto the specimens to produce minimal shrinkage during processing. Excess water was removed with a clean tissue. The specimens were then fired under vacuum to 940°C. The remaining 10 NA and BA specimens had two layers of opaque (Ceramco II) applied and fired separately under vacuum to 990°C. Subsequently, these specimens had a pressable ceramic wax-up applied using an ash-free wax (Galileo, Talladium, Valencia, CA) to a thickness of approximately 1.0 mm. The metal-opaque-wax specimen was sprued and invested in a phosphate-bonded investment (Finesse). Wax elimination was performed to 850°C (Accu-Therm II 250). PC ingots were placed in the pressing machine ring of a combi-furnace (Touch and Press 02797, Dentsply, York, PA) with a plunger on top of the ingot, and the specimens were pressed with a PC (CPC-MK, Chemichl AG, Liechtenstein) at 920°C (Table 2). The specimens were then divested and sandblasted for 15 seconds with 50 μm aluminum oxide at 60 psi. The ceramic sprue was removed with a carborundum disk. For all groups, the ceramic specimen was shaped using a polisher (Phoenix Beta) to dimensions of 8.0 mm × 1.0 mm × 3.0 mm (Fig 1).

Specimens were then subject to a three-point bending test as per ISO/FDIS 9693: 1999, on a testing machine (Model #5566, Instron, Canton, MA). The ceramic specimens were placed symmetrically on the opposite side of the applied load and equidistant between the two specimen supports which lay 20.0 mm from each other. The applied force was transmitted through a symmetrically aligned bending piston at a rate of 1.5 mm/min (Fig 2). Fracture loads (F_{fail}) were recorded in Newtons and debonding/crack initiation strengths (D/CIS) calculated via the formula $\tau_{\text{b}} = k \times F_{\text{fail}}$:

1. τ_{b} is the D/CIS and is reported in megapascals (MPa).
2. k (mm^{-2}) is a constant which is a function of the thickness of the metal specimen, d_{M} (mm), and its elastic modulus, E_{M} (GPa).
3. F_{fail} (N) is fracture load recorded by the computer at the peak MC load before debonding/crack initiation.

Digital video recordings (DCR-TRV33, Sony, Japan) were made of four randomly selected, representative samples to determine where the MC debond initiated.

Two representative samples from each of the four experimental groups were completely debonded. These test specimens exhibited a thin, transparent film of ceramic at the debonded MC interface making identification of remaining constituents difficult by visual examination only. Therefore, mode of bond failure was assessed via examination under light microscope at 35 to 100×

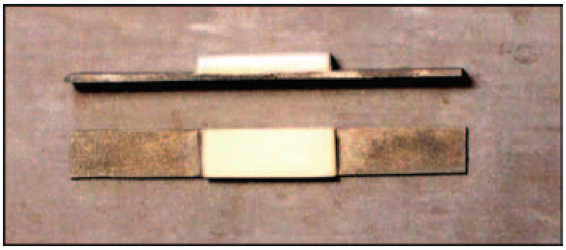

Figure 1. Prepared metal-ceramic specimens.

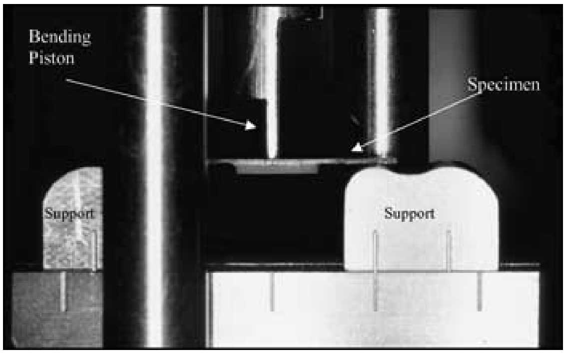

Figure 2. Mounted specimen on the testing apparatus.

magnification (Olympus SZX-ILLB100, Olympus Optical, Japan) and scanning electron micrograph (JSM 5400, Jeol, Japan) equipped with an X-ray microanalysis system (Evex, Princeton, NJ) at 200 to 500× magnification. For use of the scanning electron micrograph (SEM), specimens were carbon coated with approximately 60 nm thickness of coating (PS2, International Scientific Instruments, Australia). Subsequent topographic and elemental spectrum analysis was performed with a detector window size of 10.0 mm^2, counting levels of 1000 to 2500 counts/seconds (spectra were obtained with at least 1 minute of counting time), and an accelerating voltage of 20 kV. Analysis was done in qualitative mode.

The D/CISs between the four experimental groups were analyzed using a two-way analysis of variance (ANOVA). A one-way AVOVA was used to analyze the effect of metal specimen thickness, measured symmetrically in mm along two points of the metal specimens with a digital caliper (Series #500), on the four groups. This was done to supply d_M which is part of the formula mentioned above. A Pearson correlation was used to analyze the correlation between d_M and τ_b.

Table 3. Results

Alloy	*Ceramic*	*N*	*Mean τ_b (MPa)*	*Standard Deviation*
Noble	Feldspathic porcelain	10	32.56	4.62
Noble	Pressed ceramic	10	30.23	5.06
Base	Feldspathic porcelain	10	30.98	4.41
Base	Pressed ceramic	10	31.81	3.48

Results

Mean D/CISs, measured in MPa, with standard deviations are shown in Table 3. The two-way ANOVA ($p > 0.05$) demonstrated no significant difference between the four experimental groups (1. NA-FP, 2. NA-PC, 3. BA-FP, 4. BA-PC). Hence, type of metal and/or type of ceramic did not make a difference in τ_b.

The thickness of the metal samples, d_M, was the only observer-dependent variable. A one-way ANOVA found no significant differences between the four groups ($p > 0.05$). A Pearson correlation showed no significant correlation between d_M and τ_b ($p > 0.05$).

Digital video evaluation of specimen debonding/crack initiation showed that MC bond failure occurred at the terminal point of the MC interface (Fig 3).

SEM spectrum (200 to 500×) and light microscope (35 to 100×) analysis of the eight completely debonded specimens demonstrated that initial failure was mostly cohesive. Significant silica, aluminum, potassium, and calcium peaks were found in all SEM spectrum analyses, indicating the presence of ceramic at the debonded interface of the metal substrate. Palladium peaks were found at the debonded interfaces of the noble metal specimens, and chromium and nickel peaks were found at the debonded interfaces of the base metal specimens, indicating some metal exposure. The size of the metal peaks was not relative to either metal alloy or ceramic type. Metal peaks indicating exposure varied depending on what interface area was examined.

Discussion

The results of this study indicate that the D/CIS of a PC fused to metal was equivalent to FP fused to metal. Since most other studies in the literature were done before the ISO standard was developed, it is difficult to relate the findings to other investigations.

The magnitude of the mean D/CIS measured fell within ISO qualifications of at least four experimental specimens having a D/CIS of 25 MPa.[22] Minute differences in thickness of the metal specimens are expected during processing, but the variations exhibited in this study

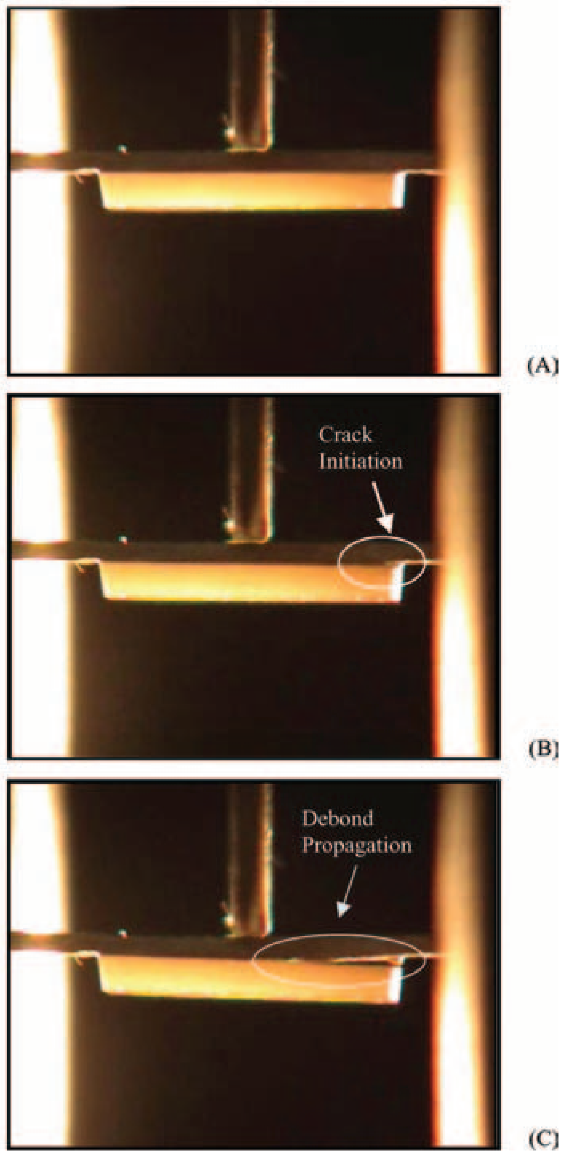

Figure 3. Sequential digital video still shots of specimen debonding/crack initiation. Note direction of debond/crack initiation from the terminal point of the specimen toward the center. (A) Time: 0 seconds. (B) Time: 8 seconds. Debonding/crack initiation occurring at the terminal end. (C) Time: 17 seconds. Debond propagation.

did not have significant bearing on the D/CISs calculated.

When comparing different groups it is critical to treat them equally. Shaping of the FP and PC after bonding to metal was performed on all groups; however, each PC specimen required divestment and that the ceramic sprue be cut off (Fig 4). In this study, there was no way to evaluate the potential impact of this procedure on the D/CIS calculated.

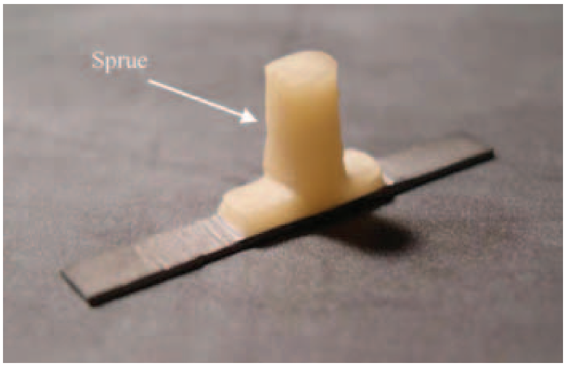

Figure 4. Unfinished metal-pressed ceramic specimen with an intact sprue.

During experimentation, all samples exhibited a progressive debond from the terminal site of one end of the MC interface toward the other. This is consistent with three-point flexural stress analysis, as reported by Anusavice et al,[23] which indicated that tensile forces on the MC bond are the greatest at the ceramic termination sites. Furthermore, SEM and light microscope examination of representative samples of all four experimental groups showed ceramic still attached to metal. Spectrum analysis demonstrated that metal was also exposed, indicating a mixed mode of failure. This is also consistent with previous three-point bending studies of MC interfaces.[20,24]

Show-through from oxide layers, and other components, refers to the fact that the bulk analysis technique used examined a volume of the sample starting with the surface of the specimen and extending inward a certain distance. Show-through refers to the concept that the reported spectrum reflects this volume and may include composition from underlying components other than what is on the surface. The amount of show-through depends on electron beam parameters (kilovoltage) and the elemental composition (atomic number and density) of the substrate. For instance, a 20 kV electron will penetrate less than 0.5 μm in gold, but may penetrate 5 μm in calcium. Based on an Evex simulation program used to estimate beam penetration, show-through from underlying materials would probably not occur through more than 2 to 3 μm of the ceramic material in question. In extremely thin areas of

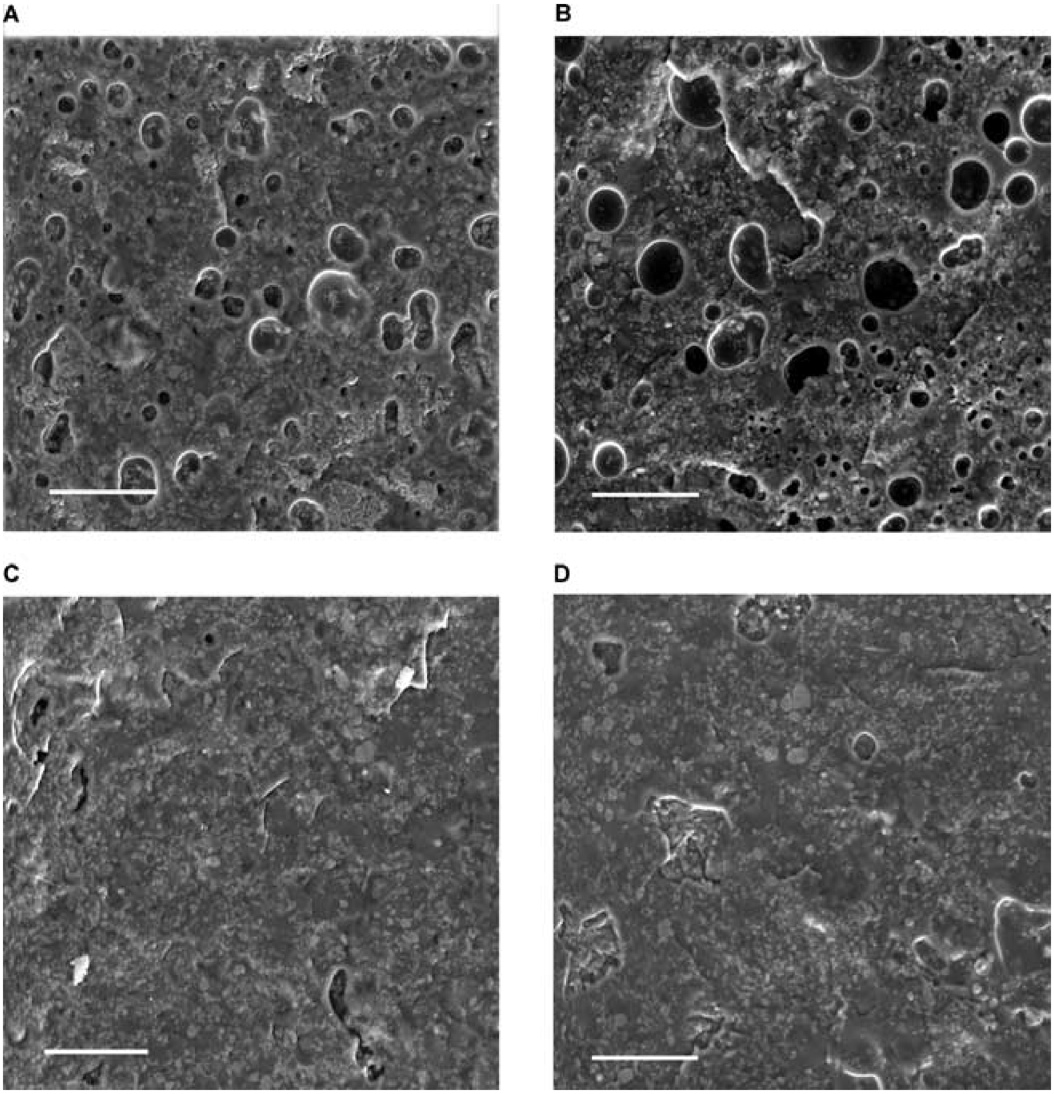

Figure 5. SEM 500× (white bar represents 50 μm in all four figures). (A) Noble alloy interface with feldspathic porcelain. (B) Noble alloy interface with pressed ceramic. (C) Base alloy interface with feldspathic porcelain. (D) Base alloy interface with pressed ceramic. Note the difference in porosities between the noble alloy specimens (A & B) and the base alloy specimens (C & D).

ceramic (less than 3 μm) one might expect to see some of the elements of the metal substrate in the spectrum.

During SEM examination, at 500× magnification, a greater incidence of porosities at the MC interface of the NA specimens was noted, although this phenomenon was not measured (Fig 5). Processing procedures, such as sandblasting and oxidation of the metal samples, resulted in a rougher surface of the NA specimens compared to the BA specimens (Fig 6), due to different physical properties of the two types of metals used, such as yield strength and elastic modulus. Carr and Brantley[25] indicated that during casting procedures, liquid palladium can absorb large quantities of gases. These gases, in turn, are released during the solidification process and must be accommodated by a coarse-grained investment material or microscopic porosities may occur in the casting. Hofstede et al contended that a rough metal surface is also capable of trapping air pockets and contaminants, which may lead to gas formation during ceramic firing, causing porosity production in the ceramic.[26] In addition, it has been reported

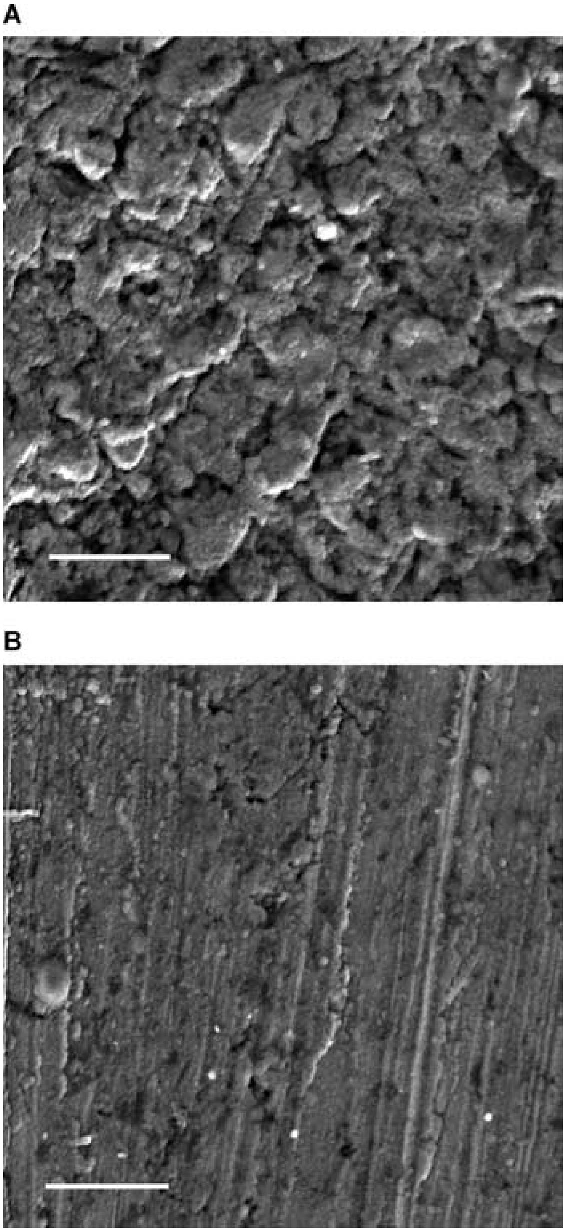

Figure 6. Metal surface. SEM 500× (white bar represents 50 μm in both figures). (**A**) Noble Alloy. (**B**) Base Alloy. Note differences in surface roughness.

that palladium oxides are unstable and dissociate at elevated temperatures, possibly creating oxygen bubbles at the MC interface.[27]

Initial bond failure, when examined under light microscope and with SEM, was found to be mostly cohesive through the body ceramic, at the terminal points of all eight completely debonded MC interfaces. This indicates that at this point the adhesive bond of ceramic to metal was greater than that of the cohesive bond of the ceramic. The bond failure then propagated to the area of greatest stress concentration at the opaque/oxide layer and continued in mixed mode toward the other terminal point of the MC specimen (Figs 7 and 8), where cohesive body failure occurred in four out of eight specimens. Considering these experimental observations, this testing configuration may be subject to limitations similar to what has been previously described in the literature.[19-21] Akin to what was reported in a study by Marques de Melo et al,[28] within the limits of this experiment, the MC bond was not tested to its limit.

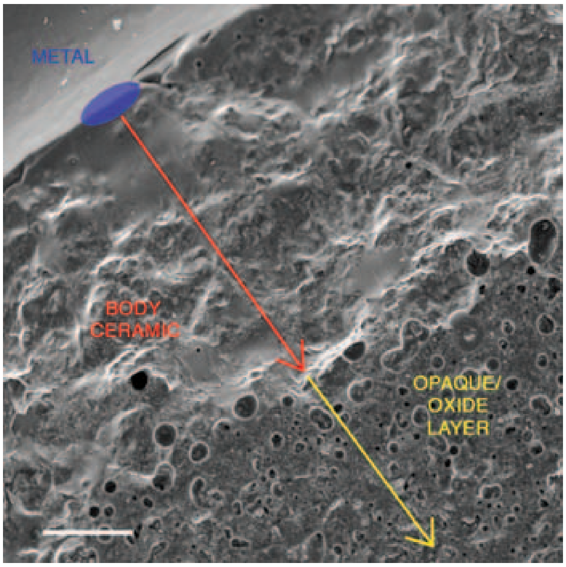

Figure 7. Terminal point of the noble alloy interface with feldspathic porcelain. SEM 200×. White bar represents 100 μm. Blue circle indicates the terminal point of the metal-ceramic interface. Arrows indicate the direction of bond failure toward the center of the specimen. Failure was initially cohesive through body ceramic and then cohesive/adhesive through the opaque/oxide layer.

The data from this study suggest the clinical potential of combining the favorable attributes of a PC with that of a metal framework. Dental materials research such as this serves as a pilot study for continued investigation. Future research should include clinical trials of a PC fused to a metal framework to determine the efficacy of such a treatment modality.

Conclusion

Under the conditions of this study ($p > 0.05$), the debonding/crack-initiation strength of a

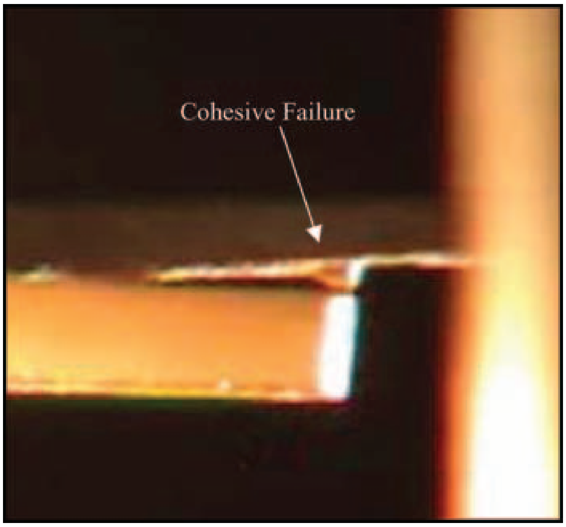

Figure 8. Macroscopic view, via digital video recording, of the cohesive bond failure, through body ceramic at the terminal point of the metal-ceramic interface.

low-fusing pressable leucite-based glass ceramic fused to metal was equivalent to that of a feldspathic porcelain fused to metal.

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