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ORIGINAL RESEARCH

Comparison of Adhesion of a Novel Pre-sintered Cobalt–Chromium to Pre-sintered Zirconia and Cast Nickel–Chromium

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ABSTRACT

Aim: This study compared the bond strength of pre-sintered Ceramill Sintron to pre-sintered zirconia and cast nickel–chromium (NiCr).

Materials and methods: Specimens (n = 60) (diameter: 15 mm; thickness: 2 mm) were prepared (n = 20/group) (Ceramill Sintron, Ceramill Zi, and Wirobond 99). Disks were layered with vita VM ceramic (4 mm). Specimens were randomly divided into two subgroups. Only one subgroup was thermocycled. Specimens were tested under shear strength. Energy-dispersive X-ray (EDX) mapping was done on one disk of each material before and after ceramic layering.

Results: Failure types were mostly mixed failures. Significant difference was found between the three materials for Y and Z failure types (p-values: 0.032 and 0.010 respectively). Thermocycling had no major effect on the results reported. Considering Fmax (force-inducing bonding failure) registered, significant difference was found between the control group and milled alloys groups. No significant difference was found between Ceramill Sintron and Zi. The EDX mapping showed a net increase in the control group oxide layer, whereas only slight increase and decrease were reported for Zi and Sintron respectively.

Conclusion: When compared with cast NiCr, novel Ceramill Sintron has higher bond strength, comparable to Ceramill Zi. Thermocycling had no major effects on the results.

Clinical significance: Ceramic–alloy bonding is a primary factor in the prosthesis' longevity.

Keywords: Computer-aided design/computer-aided manufacturing, Pre-sintered cobalt–chromium, Shear bond.

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INTRODUCTION

Although the trend in modern dentistry is to use metal-free restorations, conventional materials successfully used over many decades still keep their role as dental prostheses. Nonprecious metal alloys have proved long-term prognosis. This class of alloys has been adopted to reduce precious metal alloys' high cost. These alloys have permitted high-quality treatment for a large number of patients, unquestionably needed where framework's high strength is required.[1]

Porcelain–metal strong bond is a first requirement for long-term success of metal–ceramic restorations. It must resist both transient and residual thermal stresses and mechanical forces encountered in clinical function.[2] Core surface roughness, wetting properties, cooling rate systems, presence of flaws, residual stress arising from difference of thermal expansion and contraction between materials, and viscoelastic and elastic properties are factors that influence core–veneer interface.[3] Therefore, it is essential to focus onto metal–ceramic interfaces behavior.[4] Metals and porcelains have to be chemically, thermally, mechanically, and esthetically compatible.[5] Some degree of coefficients of thermal expansion (CTE) matching is a prerequisite.[6]

Although base-metal alloys present many superior mechanical properties, some disadvantages like poor biocompatibility, low corrosion resistance, and porcelain discoloration are often cited.[7] However, performances and properties of metal/ceramic complex are not completely defined.[8] Porcelain chipping or fracture may occur within ceramic substrate or at the metal–ceramic interface.[9] Alloy composition influences ceramic–core bond strength.[10] Air-particle abrasion with Al_2O_3 can improve shear bond strength between metal and layering ceramic.[1]

While manual casting technique was mainly used for nonprecious metal alloys processing, computer-aided design/computer-aided manufacturing (CAD/CAM) techniques were introduced. Cobalt–chromium (CoCr) frameworks were fabricated via selective laser melting or milling from fully sintered blanks. These methods needed costly investments. The two latter processing options were therefore, mainly reserved for production centers specialized in industrial fabrication of CoCr restorations.

In the early 1990s, yttrium oxide partially stabilized tetragonal zirconia polycrystal (Y-TZP) was made available to dentistry as a core material for all-ceramic restorations through CAD/CAM technique. Thanks to toughening transformation mechanism, superior mechanical properties were reported.[11] Delaminations with exposure of zirconia core[12] and minor ceramic chip-off reduce veneered zirconia long-term success.[13] Ceramill Zi (AmannGirrbach, Koblach, Austria) (AG) is designed to be milled out of porously pre-sintered zirconia ceramic blanks as enlarged constructions, then sintered to full density and shrunk to the desired final dimensions.

Recently, AG also introduced new CoCr alloy that follows the same milling process (Ceramill Sintron, AG). Dry milling is possible since this soft metal block has mechanical properties similar to those of wax block. This reduces the risk of contaminating the finished materials. This method lowers milling time as well as stress on milling machines. After milling, specimens have to be sintered at 1,280°C.[14]

The mechanical integrity and ceramic core adhesion have proven to be key factors for bilayered restorations' successful performance. Their initial bond strength and reliability after thermocycling can provide useful information of clinical behavior and predictability.

The objectives of this study were to evaluate the shear bond strength of novel pre-sintered CoCr alloy and to compare the results to pre-sintered zirconia core ceramics and conventional NiCr cast alloy with their corresponding veneering ceramics. Thermocycling effect of on shear bond strength was also investigated.

The null hypotheses were that there was no difference in shear results between tested materials. Thermocycling treatment would affect these results.

MATERIALS AND METHODS

Frameworks Fabrication

Sixty disk-shaped specimens with 15 mm diameter and 2 mm thickness were prepared (n = 60), divided into group Si: (n = 20) pre-sintered CAD/CAM CoCr (Ceramill Sintron, AG); group Zi: (n = 20) pre-sintered CAD/CAM Zirconia (Ceramill Zi, AG); and group Wi: (n = 20) NiCr lost wax technique: (control group) (Wirobond 99, Bego, GmbH, Bremen, Germany). Disks were manufactured from a disk-shaped three-dimensional body (STL file) construction, with 15 mm diameter and 2 mm thickness. Then, these specimens were nested into corresponding material blanks using Ceramill Match 2 Software (Table 1). First two groups were milled using Ceramill Motion 2 milling machine. Then, disks were cut out of blanks and sintered [Ceramill Sintron (Sintering furnace: Ceramill Argotherm, standard sintering program)] and [Ceramill Zi (sintering furnace: Ceramill Therm, standard sintering program)]. Group three wax patterns were casted in NiCr alloy following the manufacturer's instructions.

Ceramic Layering

Only one of the specimens surface was prepared for ceramic layering. NiCr disks were abraded with 150 µm aluminum oxide airborne particle (Korox, Bego) at an angle of 45° for 10 seconds from a distance of approximately 2 cm, under 2 bar pressure. Disks were then ultrasonically cleaned in isopropyl alcohol (Vitasonic II, Vita, Bad Säckingen, Germany) for 5 minutes and allowed to dry at room temperature. The CAD/CAM specimens were also layered following the manufacturer's instructions.

Ceramic layering was processed as follows: Application of two liner firings (Vita M13 and M9, Vita zahnfabrik, Bad Saeckingen) (Vita M 13 for CoCr and NiCr, Vita M9

Table 1: Disk materials and their corresponding veneering ceramic

Disks	Ceramill Sintron Lot: 1404009 n = 20	Ceramill Zi Lot: 1409001 n = 20	Ceramill Wax: n = 20 [casted in NiCr (Wirobond, Bego)] n = 20
Ceramic	(Vita M13 Vita zahnfabrik, Bad Saeckingen)	(Vita M9, Vita zahnfabrik, Bad Saeckingen)	(Vita M13, Vita zahnfabrik, Bad Saeckingen)

for Zirconia). The first ceramic dentin was layered using silicone molds, then fired in a furnace (Vita Furnace, Vita zahnfabrik, Bad Saeckingen), followed by the second layer and final glaze according to the manufacturer's instructions. Four millimeters ceramic thickness was standardized using a polyethylene template. The mold dimensions were increased to compensate porcelain firing shrinkage. Ceramics were condensed by slightly knocking the entire model (with the silicone mold) on a flat surface several times. After removal from the assembly, ceramic was fired.[15] Ceramic thickness for all the specimens was measured with a digital caliper (Mitutoyo Manufacturing Company Ltd).[6]

Fatigue Tests

Specimens (20 per group) were randomly divided into two subgroups. Only one subgroup was subjected to thermocycling. Thermocycling was performed using thermocycling device (Willytec, Gräfelfing, Germany) between 5 and 55°C for 500 times (dwell time: 30 seconds, transfer time from one bath to the other: 5 seconds). Then, the two groups were submitted to shear bond strength test.[16]

Shear Bond Strength Test

Shear bond strength tests were performed using universal testing machine (Zwick ROELL Z2.5 MA 18-1-3/7, Ulm, Germany). Load was applied with a 50 kgF load cell to substrate–adherend interface, as close as possible to substrate surface at a crosshead speed of 0.5 mm/min.[1] The stress–strain curve was analyzed with software program (TestXpert®, Zwick ROELL, Ulm, Germany). Bond strength after each test (MPa) was calculated by dividing the maximum load (N) by ceramic surface area.

Chemical Analysis of the Ceramic–Alloy Interface

Three additional specimens from each ceramic–alloy combination were prepared to be evaluated under scanning electron microscope (SEM) and EDX spectroscopy (EDS). Prior to analysis, specimens were embedded in autopolymerizing acrylic resin (Alike, Gc America Inc.), then sectioned longitudinally using diamond saw (Kerr, Orange, CA), ground finished to 400 grit silicon carbide abrasive and polished with diamond paste in the sequence of 6, 3, and 0.25 µm felt disks under water coolant irrigation (Cosmedent, Chicago, Illinois). Morphology and chemical analysis were monitored at the ceramic–alloy interface of each group using an SEM equipped with an EDS (EDAX Apollo, accelerating voltage 20 KV).

Failure Analysis

Specimens were analyzed using digital microscope (VHX-2000D; Keyence, Osaka, Japan) at 200× magnification. Failure modes have been classified into five types: X/adhesive with no opaque ceramic on metal surface, Y/opaque ceramic on substrate surface <1/3, Z/opaque ceramic on substrate surface >1/3, V/opaque ceramic completely covering substrate surface, and W/cohesive fracture into veneering ceramic.

Statistical Analysis

Statistical Package for the Social Sciences software (IBM SPSS Statistics, v23.0; IBM Corp) was used. One-way analysis of variance was used for shear bond strength values comparison between materials and for aging treatment effect. Levene's test and Bonferroni correction were performed for multiple pairwise comparisons. Chi-square test was used for failure type analysis. Two-parameter Weibull distribution values including Weibull modulus, scale (m) and shape (0) values were calculated. p-values less than 0.05 were considered significant in all tests.

RESULTS

Failure types were mostly mixed failures. Significant difference was found among the three materials failure types, for Y (p = 0.032) and Z (p = 0.010) (Table 2 and Fig. 1).

Thermocycling had no major effect on the reported results (Table 3).

Considering registered Ffail, significant difference was found between the control group and milled alloys groups. No significant difference was reported between Ceramill Sintron and Ceramill Zi (Table 4).

The EDX mapping showed a net increase in oxide layer for CoCr and NiCr groups. Only very slight increase was reported for Zi (Table 5).

The highest Weibull parameters were obtained with the NiCr group both for the Fmax and for the adhesion parameters (Graph 1).

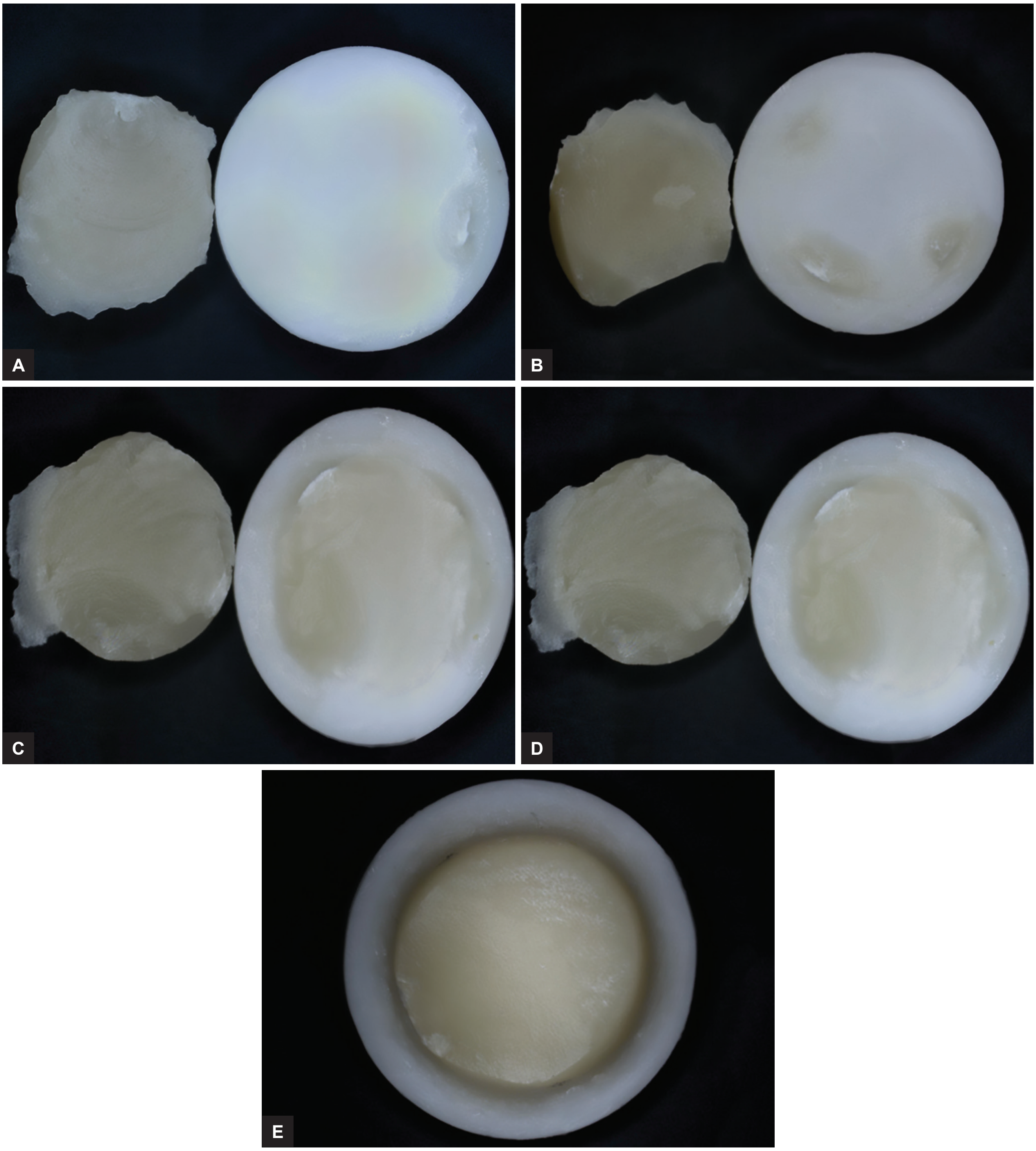
DISCUSSION

This study showed that porcelain bond strength was similar between the two milled CAD/CAM alloys. Significant difference was found with the control group.

Table 2: Percentage of type of failures for each material

Failure percentage	*X*	*Y*	*Z*	*V*	*W*	
ST	2	17	1	0	0	failure/20
	10%	85%	5%	0%	0%	
ZI	1	15	2	0	2	failure/20
	5%	75%	10%	0%	10%	
NiCr	0	9	6	3	2	failure/20
	0%	45%	30%	15%	10%	
p-value	0.0611	0.032*	0.010*	0.056	0.322	

*Significant difference



Figs 1A to E: Representative failure types on zirconia specimens. (A) Adhesive fracture with no opaque ceramic on the alloy surface; (B) opaque ceramic on the substrate surface <1/3; (C) Opaque ceramic on the subtrate surface >1/3; (D) Opaque ceramic covering the total subtrate surface; (E) Cohesive fracture in veneering ceramic

Table 3: Impact of thermocycling for each material and failure type

Shear/thermo + shear/p-value	*X*	*Y*	*Z*	*V*	*W*
ST	0.153	0.030*	0 060	0.569	0.567
ZI	1.000	0.279	0.214	0.569	0.567
NiCr	0.569	0.030*	0.538	0.014*	0.567
All groups	0.120	0.348	1.000	1.000	1.000

*Significant difference

Table 4: Mean, standard deviation, and p-value of fail between materials

Ffail/p-value	*Mean (MPa) + SD*	*p-value/all*
ST	18.446 ± (7.098)	0.105
ZI	21.973 ± (5.288)	0.350
NiCr	15.706 ± (5.879)	0.000*

*Significant difference; SD: Standard deviation

Table 5: EDX oxide mapping of the three materials with and without ceramic

EDX (Wt %)	*Disk*	*Disk + ceramic*
ST	6.17	17.12
Zi	11.92	12.88
NiCr	10.18	20.08

In accordance with other publication, shear bond strength values of Si novel material were higher than those of cast control group.[17] Null hypothesis was partly rejected.

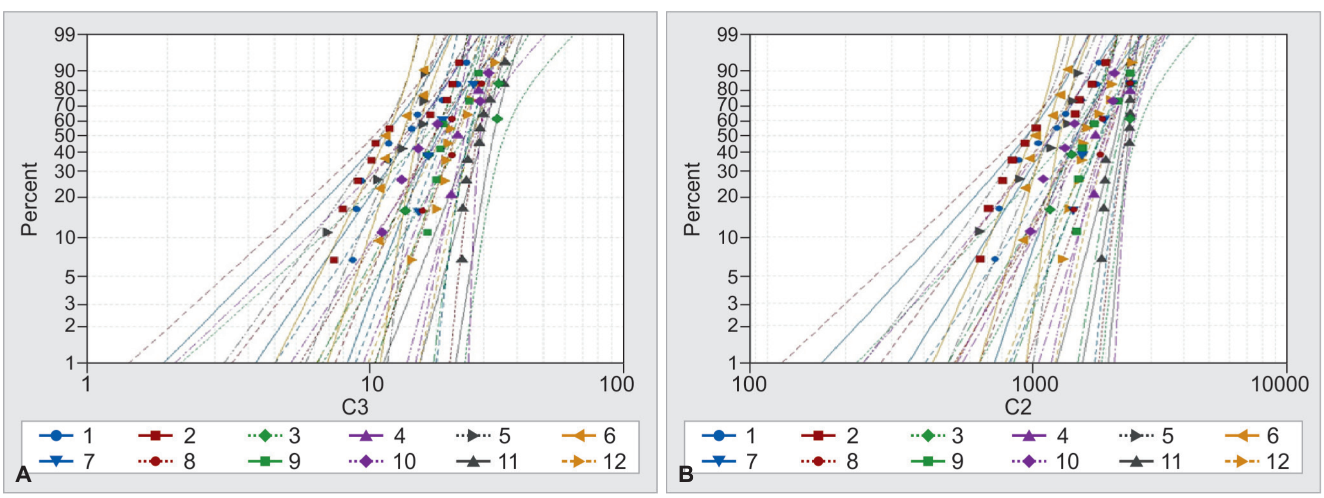
Divergent results between milled alloys and cast alloy may primarily be attributed to different adhesion mechanism of cast metal and milled core materials to veneering ceramics. Different surface topographies were reported by studies between cast and milled materials.[18] Whereas the most prominent role in the metal ceramic interface seems to be played by mechanical interlocking and chemical bond resulting from suitable metal oxidation and interdiffusion of ions,[19] bonding mechanisms of veneering ceramics to Y-TZP surfaces remain unclear.[20]

Results also reported lower values than those of other researches.[16,21] This is probably caused by differences in SBS methodology used in the current study and others. Bond strengths of smaller specimens are higher than those of larger specimens.[22] The latter show more frequent interface flaws and higher risk of early bond failure.[23] Lower values for zirconia testing, in the range of 9.4 to 12.5 MPa, were found.[11] Mean of SBS to metal and Zr was recently evaluated at 24.57 and 20.88 respectively.[24] Results of SBS test methods to various alloy types ranged from 8.458 to 97 MPa.[25,26] The SBS test method was chosen in this study due to its simplicity, ease of specimen preparation, and simple test protocol. However, some disadvantages were cited such as high standard deviations, nonuniform interfacial stresses, porosities, and specimen geometry. Although veneering process was conducted according to manufacturer's instructions, some variations can still be related to specimen preparation. A silicone mold was prepared as a hollow cylinder. Porcelain powder was packed into the tube to reproduce the same designed ceramic. This method helped to elaborate porcelain veneer form, taking shrinkage into consideration.[27] However, porcelain veneer may be damaged while removing the mold from the core. This may have decreased its bond strength.[17] The SBS is affected by veneering technique, pressed ceramic showing higher scores.[28] Ceramic layering requires good dexterity, multiple applications, and adjustments to acquire definitive shape.[29] Failure rate caused by fracture and exfoliation of porcelain was the highest reported.[30] However, heat-pressed ceramics have structural limit caused by defective interface.[31]

Weibull analysis is used to determine if the test method had a significant effect on bond test results. Test method showed influence on the study outcomes. Specimens within the same group do not fail at a single reproducible Fmax, but a distribution strength value is based on their flaw population.[32] Weibull distribution is based on the theory that the strength of a loaded body is determined by the largest structural defect.[33] A material with the highest Weibull modulus may be selected for a reliable clinical use. Si novel material can be considered for clinical applications.

It was suggested that groups should contain more than 10 specimens to obtain reliable conclusions.[34] Each group in this study was formed of 20 samples. Fractographic analysis helps to assess the critical flaw.[32] This was not the scope of the present search.

Load application technique may also have impact on the reported fail. Researcher should be aware of the well-known problems resulting from non-uniform shear stress states at the interface, related to variations in specimens



Graphs 1A and B: Probability plot with Weibull curves (95% confidence interval) using maximum likelihood estimation, scale and shape values for (A) Fmax and (B) MPA values for all groups geometry and loading configuration.[35] Specimens were designed to have an interface parallel to the load.[17] Inappropriate test design may generate false interpretation of bond strength data.[36]

Some authors proposed that all materials should be subjected to fatigue conditioning before mechanical testing.[37] In the current study, bond strengths values were irrespective of thermocycling exposure. Only significant difference was found for Sintron group, Y failure type ($p = 0.030$); and for NiCr group, failure types Y and V, (p-values 0.30 and 0.14 respectively) (Table 3). Differences in all other failure types (12 subdivisions) were nonsignificant. This confirmed results reported by others.[11] Second null hypothesis was therefore, partly rejected.

Moreover, microscope examination showed that most fractures were classified as mixed failures. Only two specimens from Sintron group and one from Ceramill Zi exhibit a full debonding of opaque from metal. This was not reported for the control group where three cases revealed a persistent opaque on substrate full surface. Previous studies reported fractures mostly adjacent to core veneer interface,[38,39] while others reported mostly cohesive failure mode in metal ceramic group.[40] 10% of the specimens showed cohesive fractures within the porcelain in groups Zi and Wi. Cohesive fracture happens when ceramic strength is less than that of ceramic core interfacial bond strength. This may be due to an inherent flaw into the porcelain. Fifteen specimens (75%) in zirconia group showed mixed debonding with less than one-third ceramic remaining on the substrate. This is in contrast with other findings that presumed that fractures in zirconia restorations were mainly chipping within the porcelain.[41] This is also applied to Ceramill Sintron where 17 specimens (85%) showed mixed debonding with less than one-third ceramic remaining on the substrate. Significant difference was found when compared with the control group where only nine specimens (45%) showed Y failure type. This is an important finding that will raise again the delamination occurrence and restorations ultimate failure for presintered milled alloys.

During firing cycle, thermo-mechanical stresses are created by differences in thermal contraction and expansion of alloy and porcelain. These transient and residual thermal stresses depend on thermal compatibility between porcelain and alloy.[42] It is agreed that alloy should have higher CTE than porcelain (a positive expansion coefficient mismatch) to produce compressive stresses into the porcelain on cooling.[5] Metal and porcelain are considered compatible if difference in CTEs is less than $1 \times 10^{-6}/°C$ at a given temperature.[43] Declared CTEs for Sintron and Wiron99 are 14.5×10^{-6} K^{-1}, $13.8–14.0 \times 10^{-6}$ K^{-1} respectively, and for Vita VM 13 $13.1–14 \times 10^{-6}$ K^{-1}. Regarding Ceramill Zi and Vita VM9, CTEs are respectively $10.4 \pm 0.5 \times 10^{-6}$ K^{-1} and $9–9.2 \times 10^{-6}$ K^{-1}.

Some authors considered that oxide layer thickness formed at materials interface decreases porcelain thermal contraction coefficient and impair adhesion.[44,45] The EDS analysis showed oxide layer increase for all materials after ceramic layering. However, oxide layer was thicker in the NiCr group. This may explain the lower shear bond strength values reported for the control group. We noticed also that there was no total delamination in the Wirobond 99 group (X 0%) (Table 2). Failure may have occurred within this thick or nonhomogenous oxide layer. In contrast to other researchers' conclusions,[45] negative correlation was found between oxide layer thickness and porcelain adhesion to metal. It was also reported that oxide layers of less than 1 to 2 nm were desirable for excellent adhesion. Thicker layers may lead to lower bond strength.[46]

Shear bond strength test remains relatively simple to implement in measuring porcelain–metal interface strength.[21] However, several factors are implicated in porcelain fracture for metal ceramic restorations. Restoration's structure, fabrication processes, technical skills, and the bond between core and veneering porcelains are to be taken into consideration.[3] If this test alone is not sufficient to predict material reliability, it may help to distinguish product A from product B,[35] and to compare a novel product to a gold standard one. In this study, novel Ceramill Sintron results were higher than those of the conventional NiCr.

CONCLUSION

From this study, it can be concluded that Novel Ceramill Si is a promising material for metal ceramic prostheses. While its results were comparable to that of Zi, they were higher than conventional cast material with no effect of thermocycling. Ceramic alloy bond needs further investigations.

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