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Influence of Alloy Microstructure on the Microshear Bond Strength of Basic Alloys to a Resin Luting Cement

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The aim of this study was to evaluate the influence of microstructure and composition of basic alloys on their microshear bond strength (µSBS) to resin luting cement. The alloys used were: Supreme Cast-V (SC), Tilite Star (TS), Wiron 99 (W9), VeraBond II (VBII), VeraBond (VB), Remanium (RM) and IPS d.SIGN 30 (IPS). Five wax patterns (13mm in diameter and 4mm height) were invested, and cast in a centrifugal casting machine for each basic alloy. The specimens were embedded in resin, polished with a SiC paper and sandblasted. After cleaning the metal surfaces, six tygon tubes (0.5 mm height and 0.75 mm in diameter) were placed on each alloy surface, the resin cement (Panavia F) was inserted, and the excess was removed before light-curing. After storage (24 h/37°C), the specimens were subjected to µSBS testing (0.5 mm/min). The data were subjected to a one-way repeated measures analysis of variance and Turkey's test (α=0.05). After polishing, their microstructures were revealed with specific conditioners. The highest µSBS (mean/standard deviation in MPa) were observed in the alloys with dendritic structure, eutectic formation or precipitation: VB (30.6/1.7), TS (29.8/0.9), SC (30.6/1.7), with the exception of IPS (31.1/0.9) which showed high µSBS but no eutectic formation. The W9 (28.1/1.5), VBII (25.9/2.0) and RM (25.9/0.9) showed the lowest µSBS and no eutectic formation. It seems that alloys with eutectic formation provide the highest µSBS values when bonded to a light-cured resin luting cement.

Key Words: dental cements, dental alloys, shear strength.

INTRODUCTION

For decades, metal-ceramic dental prostheses have been used in dentistry with good clinical performance, esthetics and durability. The aim of these restorations is to combine the fracture resistance of the metal substructure with the esthetic properties of porcelain. Noble alloys have always been the first choice for ceramometal prostheses, mainly those containing gold, palladium and platinum. However, several economic crises in the last century prevented the day-to-day use of these alloys. This led to the widespread use of basic alloys, such as nickel-chromium (Ni-Cr) and cobalt-chromium (Co-Cr), as frameworks for porcelain, due to their high mechanical strength, high modulus of elasticity and good adhesion to porcelain (1,2).

The success of any indirect restorative procedure relies on bonding indirect restorations to tooth structures. The achievement of a strong and durable adhesive bond between a metal framework and a luting agent is

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important to withstand the many and varied changes in the oral environment (3). In general, metal restorations are cemented with zinc phosphate. However, the high solubility, lack of adhesion to tooth structure and particularly the high stiffness that prevents proper load transmission to the dental structure, have shifted the focus for evaluating resin luting cements. Resin luting cements are characterized by their high fracture strength, adequate bond to the dental structure and low solubility when exposed to oral fluids (4,5).

Bond strength between resins and metals is a physicochemical phenomenon, resulting in mechanical interlocking of the cement with the metal surface and chemical interactions between the oxides present on the surface of metals and the carboxylic or phosphoric acid derivatives present in the cements (6,7).

There are great variations in the composition of basic alloys within the same classification (Co-Cr and Ni-Cr), leading to the formation of different oxides on their surfaces (2), which might alter the bond quality in some aspects (8). It is known that alterations in the composition of an alloy lead to the formation of single microstructures (9) and the generated oxides are fundamental factors in determining resistance to corrosion, biocompatibility, and bonding both to polymer and ceramic materials. In spite of this, little is known about the relationship between the microstructure of basic alloys and the bond to resin cements.

The aim of this study was to evaluate the influence of microstructure of 7 basic alloys: two Co-Cr and 5 Ni-Cr (2 with and 3 without beryllium - Be) on the microshear bond strength (μSBS) to a resin luting cement.

MATERIAL AND METHODS

Seven basic alloys for ceramometal prostheses were used in this study. VeraBond (Aalba Dental Inc., Cordelia, CA, USA), Suprem Cast V (Talladium Inc., Valencia, CA, USA), Tilite Star (Talladium Inc.), Wiron 99 (Bego, Bremen, Germany) and VeraBond II (Aalba Dental Inc., Cordelia, CA, USA) are Ni-Cr alloys while IPS d. SIGN 30 (Ivoclar Vivadent, Schaan, Liechtenstein) and Remanium (Dentaurum, Pforzheim, Germany) are Co-Cr alloys. The compositions of these alloys are shown in Table 1.

Casting

Wax disks measuring 13 mm in diameter and 4 mm high (n=5) were fabricated using a metal matrix, included in silicone rings with phosphate coating (Microfine 1600; Talladium Inc.) and cast in an electromagnetic induction machine (F. lli Manfredi, San Secondo di Pinerolo, Torin, Italy). After unmolding, the disks were airborne particle abraded with aluminum oxide (50 μm), embedded in bakelite resin and polished with silicon carbide abrasive papers of decreasing abrasiveness up

Table 1. Composition of alloys and resin cements used in this study.

Alloy	Ni	Co	Cr	Nb	Mo	Be	Si	Al	Ti
Verabond (Ni-Cr-Be)	77.95	0.45	12.6		5	1.95		2.99	0.35
Suprem Cast V (Ni-Cr-Be)	74.0	-	14.0	-	8.5	1.8	-	1.7	-
Tilite Star (Ni-Cr)	76.5	-	13.5	-	6.0	-	-	-	4.0
Wiron 99 (Ni-Cr)	65.0	-	22.5	1.0	9.5	-	1.0	2.2	0.4
VeraBond II (Ni-Cr)	77.05		12.5	4	4.25	-	0.5	2.25	0.45
d. Sign 30 (Co-Cr)	-	60.2	30.1	3.2	1	-	-	1	-
Remanium (Co-Cr)	-	65	28	-	4.5	-	1.6	-	-
Resin Cement (Panavia F 2.0)	Paste A: MDP, hydrophobic aromatic dimethacrylates, hydrophobic aliphatic dimethacrylates, hydrophilic aliphatic dimethacrylates, silanated silica filler, silanated colloidal silica, dl-camphoroquinone, initiators					Paste B: Hydrophobic aromatic dimethacrylates, hydrophobic aliphatic dimethacrylates, hydrophilic aliphatic dimethacrylates, silanated barium glass filler, initiators, accelerators, pigments			

to 2000-grit (Norton, São Paulo, SP, Brazil).

Samples were prepared in a standardized manner using 110 µm aluminum oxide for 30 s at a distance of 5 mm and pressure of 60 psi. After this, the samples were rinsed in an ultrasonic bath for 15 s to remove any excess particles. The surface was ultrasonically cleaned in isopropanol (96%) for 3 min and air-dried for 3 min (10).

Six tygon tubes (TYG-030; Small Parts Inc., Miami Lakes, FL, USA) 0.75 mm in diameter and 0.5 mm high were placed on each alloy surface and were filled with Panavia F (Kuraray Medical Inc., Tokyo, Japan) luting cement. The resin cement was applied and handled according to the manufacturers' instructions. All light-curing procedures were performed with a halogen lamp (Optilux 500; Demetron, Danbury, CT, USA) for 20 s, with a standard irradiation mode of 600 mW/cm^2. The power density of the curing device was regularly checked with a curing radiometer (Demetron).

The specimens were stored in water at 37º C for 24 h. The tygon tubes were removed with a blade and then checked under a light stereomicroscope (×10) to discard any specimens with evident air bubbles or gaps at the interface. The resin cement flash extending beyond the alloy base was also removed with a blade.

A universal testing machine (model 5565; Instron, Canton, MA, USA) was used for the µSBS test. The specimens were attached to the testing device which, in turn, was placed in the universal testing machine. A thin 0.2-mm-diameter wire (Morelli Ortodontia, São Paulo, SP, Brazil) was looped around the composite resin cylinder, around half its circumference, and gently held flush against the interface. A shear force was applied to each specimen at a crosshead speed of 0.5 mm/min until failure. The force required to produce failure was then divided by the bonded area of the vinyl tube and the bond strength values were expressed in MPa.

The µSBS values of all specimens from the same alloy were averaged for statistical purposes. The data were subjected to an one-way repeated measures analysis of variance and Tukey's test at a significance level of 5%.

Microstructure

The alloy specimens were polished with silicon carbide papers of decreasing abrasiveness (#180 to #2000; Norton) and a 0.05 µm silica colloidal solution (Struers, Rødovre, Denmark), and then etched using specific conditioners (Table 2). The prepared surfaces were examined only qualitatively using an optical stereomicroscope (HMV-2; Shimadzu, Tokyo, Japan).

RESULTS

The overall µSBS values are shown in Table 3. Statistically significant differences were observed among the groups (p = 0.0001). Verabond, Suprem Cast, Tilite Star (Ni-Cr alloys) and Co-d-SIGN 30 (Co-Cr alloy) showed the highest µSBS values, which were significantly higher than those obtained with Wiron 99, Verabond II (Ni-Cr alloys) and Remanium CD (Co-Cr alloy).

All alloys exhibited a solid solution matrix in a typical dendritic arrangement, irrespective of their composition (Ni-Cr or Co-Cr). The alloy structures are shown in Figure 1.

Figures 1A, 1B and 1C (Suprem Cast V, Tilite Star and Verabond, respectively) are identical, showing

Table 2. Solutions used to condition the alloys in this study.

Alloy	Basic composition	Etching solutions
VeraBond	Ni-Cr-Be	$C_2H_4O_2$+HNO_3 (1:1)
Suprem Cast V	Ni-Cr-Be	$C_2H_4O_2$+HNO_3 (1:1)
Tilite Star	Ni-Cr	$C_2H_4O_2$+HNO_3 (1:1)
Wiron 99	Ni-Cr	20 mL HCl+0.5 mL HNO_3+6.5 g $FeCl_3$
VeraBond II	Ni-Cr	HNO_3+HCl (1:3)
d.Sign 30	Co-Cr	HNO_3+HCl (1:3)
Remanium CD	Co-Cr	HCl+H_2O_2 (10:1)

Table 3. Shear bond strength (µSBS) means and standard deviations (MPa).

Alloy	Basic composition	µSBS
VeraBond	Ni-Cr-Be	30.64 (1.7)^{a}
Suprem Cast V	Ni-Cr-Be	28.83 (2.3)^{a,b}
Tilite Star	Ni-Cr	29.82 (0.9)^{a}
Wiron 99	Ni-Cr	28.10 (1.5)^{b,c}
VeraBond II	Ni-Cr	25.46 (2.0)^{c}
d.Sign 30	Co-Cr	31.11 (0.9)^{a}
Remanium CD	Co-Cr	25.96 (0.9)^{c}

Same letters indicate no statistically significant difference among alloys ($p<0.05$).

a dentritic structure with dendrites (white arrows) and interdendritic regions (black arrows). A "light" (white arrows) phase with an fcc structure and was etched with difficulty, has a nobler phase. On the contrary, a "dark" (black arrows) phase that was etched more easily, has a less noble phase with an hcp structure.

Figures 1D, 1E, 1F and 1G are representative images of Wiron 99, VeraBond 2, Remanium CD and d.Sign 30, respectively. These alloys are characterized by a large volume of solid solution or dendrites and a small or nonexistent interdendritic portion and precipitates.

It is worth mentioning that Figures 1E and 1F (Wiron 99 and d.Sign 30) showed a large volume of dendritic portion (white arrows) and only a few precipitates within them and at the intergranular limits (gray arrows). Figure 1F (Verabond 2) as cast, shows a large dendritic structure (white arrows) and the presence of precipitates is not visible at the intergranular limit (black arrows).

DISCUSSION

Although there is a tendency to use esthetic restorative and prosthetic materials such as direct or indirect resin composite inlays, adhesive metal-free bonded bridges (partial dentures), and all-ceramic restorations, dental casting alloys are still used in a variety of dental applications. The basic alloys are of interest for conducting studies due to their low cost. However, the interaction of these alloys with luting agents is still not clear, particularly those known as resinous products.

There are innumerable factors that will influence the bond strength of a resin cement to a metal alloy, among which the following are outstanding: alloy (8,11) and cement (12) compositions, treatments to which the part is subjected, use of agents to improve bond durability (5) and cleaning of the part (10,13).

With regard to the composition of Ni-Cr alloys, there are still scarce published data (11,13). However, there are two clear reasons for the high bond strength values of these alloys: the presence of Be (14) and the high chromium content in the Ni-Cr-based alloys (8).

Be is added to reduce the melting point of alloys and thus improve castability, due to the formation of the eutectic Ni-Be phases at low melting temperature (15). Another feature associated with Be is the formation of oxide films in the inner parts of the interdendritic eutectic Ni-Be phases or on the dendrites, which provides a strong oxide that results in excellent bonding (12). The

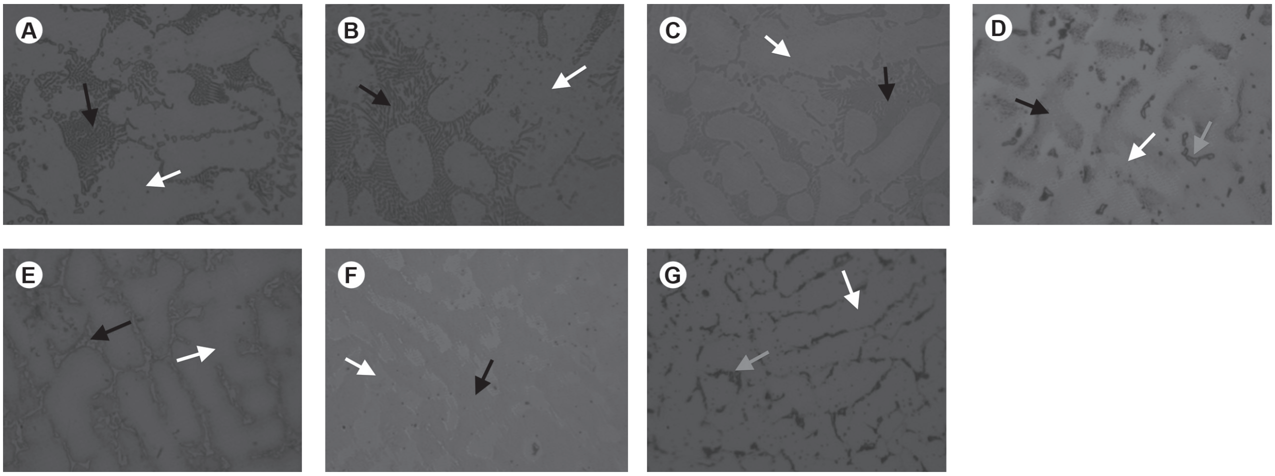

Figure 1. Optical micrographs showing the structure of the alloys: A: Supreme Cast V alloy (Ni-Cr-Be) - Dendrites (white arrows) and interdendritic region (black arrows) probably eutectic Ni-Be. B: Tilite Star alloy (Ni-Cr) - Dendrites (white arrows) and interdendritic region (black arrows). C: VeraBond alloy (Ni-Cr-Be) - Dendrites (white arrows) and interdendritic region (black arrows) probably eutectic Ni-Be. D: Wiron 99 alloy (Ni-Cr) - Large volume of dendritic portion (white arrows) and only some precipitation (gray arrows) inside and on the intergranular limits (black arrows). E: VeraBond II alloy (Ni-Cr) - Large volume of dendritic portion (white arrows) and on the intergranular limits (black arrows). It is not possible to verify the presence of precipitates. F: Remanium alloy (Co-Cr) - Alloy is characterized by the large volume of solid or dendritic solutions (white arrows) and interdendritic portion (black arrows), an alloy that is difficult to characterize due to the Cr high content and areas of high surface energy. G: d.Sign 30 alloy (Co-Cr) - Alloy is characterized by the large volume of solid or dendritic solution (white arrows) and a large volume of precipitates located at the intergranular limits (gray arrows).

interdendritic eutectic Ni-Be is the component with the highest free energy (16), which favors the bond to other materials (ceramic or resin). This becomes evident when the bond strength values of the alloys containing Be are observed (Suprem Cast V and VeraBond), confirming the microstructure analysis of the alloys. It is possible to observe the typical lamellar formation of eutectic Ni-Be on the interdendritic portion of these alloys, confirming the findings in the literature (9).

Another alloy that showed good results was Tilite Star. Although the manufacturer of this alloy claimed that there is no Be in its composition, the microstructure analysis of Tilite Star shows evidence of lamellar eutectics in its interdendritic portion, very similar to those in the Supreme Cast V and VeraBond alloys. This is an indication that the Tilite Star alloy does perhaps have Be in its composition, which would help explaining its high bond strength values.

In spite of the excellent results of Ni-Cr alloys containing Be in bond strength tests with ceramic and metal materials, this alloy causes some mistrust in the scientific community because of biological problems related to the element Be. Despite its usefulness, a number of standard setting agencies have determined that Be is a carcinogen (17). Therefore, in the opinion of some researchers, these alloys should not be used clinically (18,19). Some Be-free Ni-Cr alloys present a high Cr content. Similarly, chromium leads to the formation of an oxide layer that facilitates the chemical interaction of the cement with the alloy, which has been demonstrated in previous studies (8,20). This was indeed confirmed in the present study. The Wiron 99 alloy, which contains approximately 22.9% Cr, showed higher bond strength value when compared with VeraBond II alloy that contains only 12.5% Cr.

Panavia F 2.0 resin cement contains MDP monomer (10-Methacryloyloxydecyl dihydrogen phosphate), which can react chemically with the chromium oxide layer created on the cast metal surfaces (21,22) by means of covalent bonds (13). In the present study, the alloy primer was not used, in spite of being indicated by the manufacturer for application on the metal surface before cementation of the resin material. Various studies have demonstrated that basic alloys oxidize more easily and do not need a primer, and that the use of metal primers on basic alloys may reduce the bond strength values (3,5).

For the Co-Cr alloys assessed in this study, the Cr content does not seem to be related to bond strength. In spite of the very close percentages of Cr concentrations in the d.Sign 30 (30.1%) and Remanium CD (28%) alloys, these materials showed discrepant results of bond strength to resin cement. In the Remanium CD alloy, it was not possible to distinguish an interdendritic portion and the formation of intermetallic composites and precipitates, leaving the impression that this is an alloy with a solid single phase structure with possibly complete miscibility of the components. Whereas the microstructure of d.Sign 30 alloy shows the formation of an intermetallic composite probably rich in Nb (23) or a Co-Al precipitate (Fig. 1G - gray arrows). This may explain the great difference between the values of these two Co-Cr alloys, because of the formation of an interdendritic portion seems to favor the attainment of higher bond strength values, since there is greater surface energy in this region (16).

When this is compared with the results of the Ni-Cr alloys containing Be and one of the Co-Cr alloys, the values are very close. However, the use of alloys that contain Ni in their composition is constantly questioned due to their allergenic potential. Therefore, Co-Cr alloys are an excellent alternative to the problematic Ni-Cr alloys.

The presence of eutectic formation or the presence of precipitates in the microstructure of Ni-Cr and Co-Cr alloys appears to contribute to the bond to resin cements. Clinically, one may predict that using these alloys with eutectic formation, there is a lower risk of loss of restoration with metallic frameworks.

The presence of innumerable constituents in the composition of Ni-Cr (24) and Co-Cr (25) alloys makes it difficult to identify the precipitates forming after casting. In various figures of the present study, it is possible to observe the presence of precipitates. However, due to their very small size, it is unfeasible to identify them and thereby support their relationship with bonding. Another factor that may influence the results is the airborne particle abrasion used before the resin cement. Due to the different microhardness values of the alloys, the resultant roughness of this airborne particle abrasion may be different for each type of alloy. However, several studies have shown that some of these alloys have very close microhardness values (9,24,25) and in the present study, different bond strength values were found with the same resin cement.

The presence of eutectic formation or the presence of precipitates in the microstructure of Ni-Cr and Co-Cr alloys appears to contribute to the bond to resin cements.

RESUMO

O objetivo deste estudo foi avaliar a influência da microestrutura e da composição de ligas básicas quanto à resistência de união (RU) a um agente cimentante resinoso. Foram usadas as seguintes ligas: Supreme Cast-V (SC), Tilite Star (TS), Wiron 99 (W9), VeraBond II (VBII), VeraBond (VB), Remanium (RM) and IPS d.SIGN 30 (IPS). Cinco padrões em cera (13 mm de diâmetro e 4 mm de altura) para cada uma das ligas básicas foram incluídos e fundidos em uma máquina de fundição. As amostras foram então embutidas em resina, polidas com lixas e jateadas. Após a limpeza das superfícies, seis cilindros de tygon tube (0,5 mm de altura e 0,75 mm de diâmetro) foram colocados sobre a superfície da liga e preenchida com cimento resinoso (Panavia F), o excesso foi removido antes da fotoativação. Após armazenagem (24 h/37°C), cada amostra foi ensaiada por microcisalhamento a 0,5 mm/min. Os dados foram tratados por análise de variância para e medidas repetidas e teste Tukey (α=0,05). Para avaliar a microesturutura, as mesmas amostras foram polidas e condicionadas com solução específica. Os valores mais altos da RU (média/desvio padrão em MPa) foram encontrados para as ligas com estrutura dendrítica, formação eutética e precipitados: VB (30,6/1,7), TS (29,8/0,9), SC (30,6/1,7), com exceção da IPS (31,1/0,9) que apresentou alto valor de RU, mas sem formação eutética. As ligas W9 (28,1/1,5), VBII (25,9/2,0) and RM (25,9/0,9) apresentaram os menores valores de RU e ausência de formação eutética. Pode-se inferir que os valores mais elevados de resistência de união ao cimento resinoso fotoativado ocorre com as ligas que apresentam formação de eutético.

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