# COMPARISON OF BOND STRENGTH OF COMMERCIALLY PURE TITANIUM AND NICKEL CHROMIUM ALLOY WITH THREE DIFFERENT LUTING CEMENTS: AN IN-VITRO STUDY

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## ABSTRACT

## BACKGROUND

Metal ceramic fixed dental prosthesis remains widely used for oral rehabilitation. The type of alloy used to fabricate the metal substructure of the crown also affects its retention. The aim of this study is to compare the bond strength of commercially pure titanium and nickel chromium plates cemented with three different cements and to comparatively evaluate the bond strength of each luting cement.

## METHODS

Specimens of each metal were divided into three groups, which received one of the following luting techniques: Group 1 (CPTi) and Group 2 (NiCr) with resin cement; Group 3 (CPTi) and Group 4 (NiCr) with Glass Ionomer Cement; Group 5 (CPTi) and Group 6 (NiCr) with Zinc phosphate cement. The bonded specimens were submitted for the bond strength tests conducted with a Universal Testing Machine with a shear mode under a crosshead speed of 0.5 mm/min. Debonded specimens were examined under electron microscope.

## RESULT

The results indicate that Group 1 and 2 have significantly higher values than Group 3, 4, 5 and 6. Also, Group 3 and 4 have significantly higher values when compared to Group 5 and 6. Whereas, there was no significant difference between Group 1 and 2, Group 3 and 4 as well as Group 5 and 6. The scanning electron microscope illustrated the different modes of fracture that occurred at the metal cement interface. Resin cement showed predominantly cohesive failure. Glass ionomer cement showed a mixed mode of both cohesive and adhesive fracture and Zinc phosphate cement also showed mixed mode of fracture with predominantly adhesive failure.

## CONCLUSIONS

Resin cements showed the most superior bond with both commercially pure titanium and nickel chromium metal. Zinc phosphate cement showed the lowest bond strength with both the metals. There was no significant difference observed between the cement bond with different metals.

#### **KEYWORDS**

Bond Strength, Shear Bond, Base Metal, Alloy, Luting Cements.

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#### INTRODUCTION

Dental luting cements are the link between indirectly fabricated restorations and the prepared tooth structure.<sup>1</sup> Bonding between a metal framework and a luting cement is important to withstand the varied changes in the oral environment.<sup>2</sup> subjected to a combination of masticatory forces repeated over a period of time.<sup>3</sup> Clinicians may choose among several alloy types, surface treatments to be applied to the intaglio surface of the restoration, and must also select the most appropriate cementing agent, to maximize restoration longevity.<sup>4</sup>

Financial or Other, Competing Interest: None. Submission 20-04-2016, Peer Review 13-05-2016, Acceptance 20-05-2016, Published 06-06-2016. Corresponding Author: Dr. Lakshmi Ramesh, Door No. 03-31-2673, Next to Krishna Nursing Home Pinto's Lane, Karangalpady, Mangalore-575003. E-mail: lakshmiremesh.nambiar@gmail.com DOI: 10.14260/jemds/2016/661 Titanium and titanium alloys are being increasingly used as an alternative material to make fixed prostheses, because of their excellent biocompatibility, corrosion resistance, low density, light weight, physical and mechanical properties.<sup>5,6</sup> It is a preferred metal for fabricating the prosthetic superstructure of an implant abutment since, use of different dental alloys for the superstructure of the implants leads to detectable galvanic corrosion.<sup>5</sup>

Two important interfaces affect the ultimate bonding potential for a restoration to an abutment: Between the abutment and the adhesive cement and that between the cement and the intaglio surface of the prosthesis.<sup>4</sup> There are several factors that influence the retention-strength in cement-retained restorations including the properties of luting cements, surface area of the crown or the implant abutment and surface finish or roughness.

The other factors are height of the abutment, relative adaptation of the restoration to the abutment, strength properties of the cast metal superstructure, cementation technique, variation in cement viscosity and occlusal convergence.<sup>5</sup>

Various cements have been used to cement fixed restorations including zinc phosphate, glass ionomer and composite resin cements.<sup>6</sup> The longevity of metal ceramic restorations depends on the accuracy of all steps of treatment including their cementation. This step deserves special attention, especially when the prepared tooth has limited resistance and retention form. Little direct information is available regarding the effect of alloy selection on the strength and integrity of the metal-cement interface.<sup>4</sup> The bond at the restorative material/cement interface should rely on micromechanical retention in conjunction with chemical bonding. Airborne-particle abrasion with aluminium oxide particles is the most widely used method for providing micromechanical retention.<sup>7</sup>

These studies were encouraged by the attractive biologic, physical, chemical and mechanical properties of titanium as well as the poor bonding between cast titanium and luting materials in comparison to the commonly used nickel chromium.<sup>2</sup>

The objectives of this study are: To compare the bond strength of commercially pure titanium and nickel chromium plates cemented with Resin cement (Panavia F), Glass Ionomer Cement (GC Gold Label) and Zinc Phosphate cement (Harvard cement) and to comparatively evaluate the bond strength of commercially pure titanium and nickel chromium with each luting cement quantitatively with the universal testing machine and qualitatively observed under the scanning electron microscope and determine which luting cement provides the best bond strength with each metal and also which metal provides a better metal-cement bond.

#### MATERIAL AND METHODS

A custom made rectangular, stainless steel die (Fig. 1) was fabricated, 10 cms in length, 3 cms in breadth and 1 cm in thickness. This block had four slots with the dimensions,  $1 \times 1 \times 0.2$  cms. A counter die of the same dimensions was made  $(10 \times 3 \times 1 \text{ cm})$ .

#### **Preparation of Resin Patterns**

Prior to fabrication of each sample, the stainless steel die (Fig. 1) was coated with wax separator (Han Dae Chemical Co., Ltd). One coat of the separating medium was applied only in the slots. The slots were then filled with pattern resin (GC Corp). The counter die was placed over it and the pattern resin was allowed to set and then was removed.

#### **Fabrication of Metal Specimens**

Seventy two pattern resin samples (Fig. 2) were fabricated (1x1x0.2 cm). Thirty two samples were sprued and invested with Titec investment material (Titec; Orotig) and Commercially pure grade 2 Titanium was used for casting. The other 36 samples were sprued and invested using phosphate bonded investment (Bellasun, Bego, Germany). Nickel chromium alloy pellets (Bellabond plus, Bego, Germany) were used for casting. After casting the ring was bench cooled for 1 hour and the plates were divested using sandblasting unit (Duostar, Bego) for air borne particle abrasion with 250  $\mu m$ aluminium oxide at 60 bar pressure. The specimens were separated using carborundum disc. Each test specimen is trimmed with a hand piece and treated with air borne particle abrasion and cleaned using an ultrasonic cleaner (Sm 200lUs, India). The airborne-particle-abrasion pre-treatment for the metal surface consisted of 50-µm aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) applied at 0.3 MPa pressure for 10 seconds and then cleaned with a steam jet for 10 seconds.  $^{\rm 8}$ 

Specimens of each metal were divided into three groups (n=12), which received one of the following luting techniques: Group 1: CPTi with resin cement (Panavia F); Group 2: NiCr with resin cement (Panavia F); Group 3: CPTi with GIC (GC Gold Label); Group 4: NiCr with GIC (GC Gold Label); Group 5: CPTi with Zinc phosphate cement (Harvard cement) and Group 6: NiCr with Zinc phosphate cement (Harvard cement).

The treated commercially pure titanium (Fig. 3a) and nickel chromium samples (Fig. 3b) (10 mm x 10 mm x 2 mm) were mounted on a custom made acrylic block (Fig. 4 and 5). A Teflon block (25 mm diameter and 7 mm high) with a central cylindrical through hole (6 mm diameter and 4 mm high) (Fig. 6) was mounted on the metal surface (Fig. 7). Different luting cements were mixed accordingly and immediately dispensed into the through hole of the Teflon block up to 4 mm height and allowed to set. Group 1 and 2 received Resin cement; Group 3 and 4 received Glass Ionomer type I cement and Group 5 and 6 received Zinc Phosphate cement). Forty minutes after preparation, after the cements had completely set, the Teflon block was carefully removed (Fig. 8).

All specimens were stored in distilled water at  $37^{\circ}$ C (Precision TM, Germany) for 24 hours and then thermocycled (LAM Tech. LTC 100, Italy). The thermocycling consisted of 1000 cycles between  $5^{\circ}$ C and  $55^{\circ}$ C with a dwell time of 30 seconds in each temperature. After thermocycling, the specimens were stored in  $37^{\circ}$ C distilled water for an additional 24 hours before debonding.<sup>2</sup>

## Bond Strength Analysis

Bonded specimens were submitted to test for bond strength conducted with a Universal Testing Machine (Instron 3366, U.S.A) with a shear mode under a crosshead speed of 0.5 mm/min (Fig. 9). The values obtained by the universal testing machine were analysed using a one-way ANOVA, Tukey's HSD test and student's unpaired 't' test.

#### Scanning Electron Microscope Analysis

In order to perform a qualitative micromorphologic examination of the metal cement interphase, one representative of debonded specimen (Fig. 10) from each group was selected and examined at 200x magnification using a scanning electron microscope (Merlin, ZEISS, Germany). Failure mode was recorded by a single calibrated observer as either adhesive (Failure at the substrate-cement interface), cohesive (Failure within the substrate or within the restorative material) or combination (Areas of adhesive and cohesive failure). For this classification, the adhesive area was divided into quadrants and in each of them the predominant type of fracture was observed. The fracture was classified as adhesive or cohesive if either of these types predominated in three or more quadrants, and classified as a combination if two quadrants presented adhesive failure and the other two cohesive failure. Three variables and their interactions were investigated.2

## RESULTS

The results indicate that Group 1 and 2 have significantly higher values than Group 3, 4, 5 and 6. Concomitantly, Group 3 and 4 have significantly higher values when compared to Group 5 and 6. Whereas, there was no significant difference

seen between Group (1 and 2), Group (3 and 4) as well as Group (5 and 6).

## **Statistical Analysis**

The highest and lowest shear bond strength values in (mpa) of resin cement (Panavia F 2.0); Glass ionomer cement Type I (GC Gold Label) and zinc phosphate cement (Harvard cement) obtained for the two metals are illustrated in Table 1.

Mean and standard deviations were determined for shear bond strength of the samples of each study group as seen in Table 2. The data was analysed by use of t-test for equality of means. There was no significant P value observed in any metal category with the respective cements. Graph 1 represents shear bond strength (MPa) comparing the two metals. The one-way ANOVA test compared the three cements in two metal groups (Table 3). There was a significant difference between the cements in both metals. Graph 2 represents the shear bond strength comparing three different cements. The Tukey Honestly Significantly Different (HSD) tests (Table 4) showed multiple comparisons of cements in both metals. Dependant variable for the analysis is the bond strength between the metal and cement.

According to this study conducted, resin cements showed the most superior bond with both commercially pure titanium and nickel chromium metal. GIC was intermediate between resin cement and zinc phosphate followed by zinc phosphate cement showing the lowest bond strength with both the metals. There was no significant difference seen between the cement bond with CpTi base metal and NiCr alloy.

## The Scanning Electron Microscope

Photomicrographs illustrated the different modes of fracture that occurred at the metal cement interface. Group 1 (Fig. 11) and 2 (Fig. 12) for resin cement showed prevalence in cohesive failure. Group 3 (Fig. 13) and 4 (Fig. 14) for glass ionomer cement exhibited a mixed mode of fracture of predominantly cohesive failure and Group 5 (Fig. 15) and 6 (Fig. 16) for zinc phosphate cement demonstrated a mixed mode of fracture with predominantly adhesive failure.

Cement	Metal	Highest value	Lowest value
Resin Cement	СрТі	244.96	197.30
	NiCr	236.92	195.11
	СрТі	168.07	139.93
Glass lonomer	NiCr	166.92	134.14
Zinc Phosphate	СрТі	85.47	64.92
	NiCr	87.44	64.72

Table 1: Highest and Lowest Shear Bond Strength Values in (MPa) of Resin Cement; Glass Ionomer Cement Type I and Zinc Phosphate Cement Obtained for the Two Metals

	Metal	Ν	Mean	Std. Deviation	T-Test for Equality of Means	0	P value	
Resin Cement	СрТі	12	213	13.15539	-0.698	22	0.492	
	NiCr	12	216.7208	12.94925				
Glass Ionomer	СрТі	12	155.2058	9.70862	0.574	22	0.572	
	NiCr	12	152.7317	11.35547				
Zinc Phosphate	СрТі	12	72.54667	7.388773	-1.107	22	0.28	
	NiCr	12	75.73083	6.678851				
Table 2: Comparison of Bond Strength of Each Cement with Different Metals								

Metal		N	Mean	Std. Deviation	Mean Square	F	Sig.
CPTI	Resin Cement	12	213	13.15539	59799.685	557.286	<0.001
	Glass Ionomer	12	155.2058	9.70862			
	Zinc Phosphate	12	72.54667	7.388773			
	Total	36	146.9175	59.31525			
NICR	Resin Cement	12	216.7208	12.94925		525.768	<0.001
	Glass Ionomer	12	152.7317	11.35547	59803.844		
	Zinc Phosphate	12	75.73083	6.678851			
	Total	36	148.3944	59.36841			
Table 3: One-Way ANOVA Comparing the Three Cements in Two Metal Groups							

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Metal	(i) Cement	(j) Cement	(I-J) Mean Difference	Std. Error	Sig.		
	Resin Cement	Glass ionomer 57.7941667		4.2289718	< 0.001		
CPTI		Zinc phosphate	140.4533333	4.2289718	< 0.001		
	GIC	Zinc phosphate	82.6591667	4.2289718	< 0.001		
NICR	Desin Coment	Glass Ionomer	63.9891667	4.3540325	< 0.001		
	Keshi Cement	Zinc Phosphate	140.9900000	4.3540325	< 0.001		
	Gic	Zinc Phosphate	77.0008333	4.3540325	< 0.001		
Table 4: Post Hoc Analysis showing Multiple Comparisons of Cements in Both Metals (Tukey HSD)							



Graph 1: Graphic Representation of Shear Bond Strength (MPa) Comparing Two Metals



Graph 2: Graphic Representation of Shear Bond Strength Comparing Three Different Cements



Fig. 1: Customized Stainless Steel Die



Fig. 2: Pattern Resin Samples



Fig. 3: Samples after Sandblasting (a) Commercially Pure Titanium; (b) Nickel Chromium

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Fig. 4: Custom Made Acrylic Block



Fig. 5: Metal Plate Embedded in Acrylic Block



Fig. 6: Customized Teflon Block with a Central Cylindrical Through Hole



Fig. 8: CpTi/Ni-Cr Plate Cemented with Luting Agent



Fig. 9: Universal Testing Machine to Test Shear Bond Strength of the Samples



Fig. 7: Teflon Block Placed on Metal Plate Embedded in Acrylic Block



Fig. 10: Debonded Specimen



Fig. 11: SEM Photomicrograph of Group 1 with Resin Cement Interface Illustrating Cohesive Failure



Fig. 12: SEM Photomicrograph Group 2 with Resin Cement Interface Illustrating Cohesive Failure



Fig. 13: SEM Photomicrograph of Group 3 with Glass Ionomer Cement Interface Illustrating Mixed Mode of Fracture with Predominantly Cohesive Failure DISCUSSION

Metal ceramic fixed dental prosthesis remains widely used for oral rehabilitation. The type of alloy used to fabricate the metal substructure of the crown also affects its retention. Base metals are known to have higher free-surface energy and are more reactive than noble and high noble alloys forming a thicker oxide layer.<sup>5</sup>

The surfaces can be airborne-particle abraded to provide for a clean and roughened surface prior to cementation.<sup>1</sup> Studies conducted by Egoshi et al,<sup>9</sup> showed cement bond strength was found to be high, especially after airborne-



Fig. 14: SEM Photomicrograph of Group 4 with Glass Ionomer Cement Interface Illustrating Mixed Mode of Fracture with Predominantly Cohesive Failure



Fig. 15: SEM photomicrograph of Group 5 with Zinc Phosphate Cement Interface Illustrating Mixed Mode of Fracture with Predominantly Adhesive Failure



Fig. 16: SEM Photomicrograph of Group 6 with Zinc Phosphate Cement Interface Illustrating Mixed Mode of Fracture with Predominantly Adhesive Failure

particle abrasion to base metal alloys. According to previous studies.<sup>10,11</sup> conducted, air-abrading with 50  $\mu$ m aluminium oxide particles creates undercuts and deposits particles of aluminium on the surface of the metal, which generates physical-chemical alterations on the titanium surfaces.

Zinc phosphate cement, exhibits adequate film thickness to comply with American Dental Association.

Zinc phosphate does not chemically bond to any substrate and provides a retentive seal by mechanical means only.

The glass ionomer cement exhibit higher compressive strengths than zinc phosphate cement. Previous studies.<sup>12,13</sup>

have reported that glass ionomer cements possess low film thickness and maintain relatively constant viscosity for a short time after mixing. The main drawbacks of this cement are its well-documented susceptibility to moisture attack and subsequent solubility when exposed to water during the initial setting period.<sup>14</sup> Resin cements demonstrate good bond strengths due to micromechanical retention. They show strong adhesion as a result of chemical interaction of the resin with an oxide layer on the metal surface.<sup>14</sup>

A positive hypothesis was attained with regard to the cements demonstrating significant difference in bond strength values among each of the three types of cements, wherein resin cements showing the highest values followed by glass ionomer cement and zinc phosphate cement with lowest values. These results were similar to study conducted by Burger et al<sup>15</sup> on bond strength of different luting agents to a NiCr alloy.

Ergin et al<sup>3</sup> showed that the adhesive and mechanical properties of luting cements were highly affected by the existence of humidity and thermal effects. Hence, it is vital to consider the existence of humidity in the oral environment, in in-vitro testing of luting cement retention. Studies.<sup>16,17,18</sup> have been conducted to observe the effect of thermocycling on the bond strength of the cements with metal. They proved to be a negative influence on the bond strength values between the titanium alloy and the resin cement.

Miller et al<sup>18</sup> and Sen D et al<sup>19</sup> conducted studies concerning the bond strength of Panavia F for base metal and noble metal alloys. They elicited that base metals demonstrated significantly higher bond strengths when compared to noble metal alloys. Ergin et al<sup>3</sup> conducted a study comparing the tensile bond strength between different luting cements for base and noble metal copings. They proved that zinc phosphate cements (Phosphate) showed higher bond strength with base metal alloy (NiCr) when compared to noble metal (AuAgPd). But the results proved otherwise for glass ionomer cement (Meron) and resin cement (Avanto).

The results of this study did not support the first hypotheses that anticipated higher bond strength when using the base metal alloy. The test data obtained in this study indicated that there was no significant difference between shear bond strength of commercially pure titanium and nickel chromium with each of the cements. Other studies.<sup>4</sup> proved that metal type did not significantly affect tensile bond strength. However, alloy type and surface treatment affected site of debond location.

Abreu et al<sup>4</sup> conducted a study where differences in failure site incidences were found to be related to metal type. The noble alloy tended to fail at the metal-cement interface and within the cement itself with predominantly adhesive failure.<sup>20</sup>, while base metal did not show any particular tendency toward a specific failure location of a mixed failure for noble metals. The glass ionomer cement showed mixed type of fracture with predominantly cohesive failure, whereas for zinc phosphate cement the bond is completely dependent on the micromechanical retention.<sup>14</sup> Hence, a predominantly adhesive mode of failure was seen at the metal cement interface. This study demonstrated no difference in the mode of fracture with different metals.

In spite of several limitations, this in-vitro study suggested that the bond strength of the two metals with three different luting cements can be considered to relate directly to the clinical situation when standardized crown preparations and methods during specimen fabrication and testing are used.

## CONCLUSION

## Within the Limitations of this In-Vitro Study, the following Conclusions Were Drawn

- 1. There was significant difference between the bond strength of the three luting cements. Resin cements showed the most superior bond with both commercially pure titanium and nickel chromium metal. Glass ionomer cement was intermediate between resin cement and zinc phosphate.
- 2. The scanning electron microscope illustrated that resin cement showed a cohesive mode of failure. Glass ionomer cement showed a mixed mode of fracture of predominantly cohesive failure and zinc phosphate cement showed mixed mode of fracture with predominantly adhesive failure.
- 3. There was no significant difference seen between the cement bond with the two metals.

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