ORIGINAL RESEARCH

In-vitro investigation to evaluate the flexural bond strengths of three commercially available ultra low fusing ceramic systems to Grade II Titanium

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ABSTRACT

Background: This in-vitro investigation was designed to compare the flexural bond strengths of three commercially available ultra low fusing ceramic systems to Grade II Titanium and evaluate the type of bond failure through Scanning Electron Microscopy (SEM) and Energy Dispersion X ray Spectrum (EDS).

Materials & Methods: Sixty patterns of auto polymerizing resin of dimensions 25.1 mm X 3.1mm X 0.6mm each were fabricated from a stainless steel die. Titanium casting for all the samples was done in a Titanium casting machine. Ten samples were allotted to each of three groups randomly. Ceramic build up was done step by step using the manufacturers' instructions. Flexural Bond strength tests for the samples were performed by using a 3-point bending test on a Universal testing machine in compliance with Deutsches Institut für Normung (DIN) 13.927 draft. After the tests were completed, three samples, one from each group were selected randomly for the subsequent Scanning Electronic Microscopy (SEM) studies to characterize the type and morphology of the fracture in representative specimens.

Results: The maximum load to fracture the porcelain was recorded for each specimen. All the means of the three groups were compared by one-way Analysis of Variance (ANOVA) and it was found that Group I & Group III had significantly higher bond strength values as compared with the Group II ($P < 0.001$). All the 10 samples of Group I & III gave bond strength values above the standard values of 25 MPa. There was statistically significant difference in the bond strengths between Group I & Group III ($t = 2.76$ and $P < 0.05$), between Group I & II ($t = 5.09$ and $P < 0.001$) and between Group II & Group III ($t = 13.28$ and $P < 0.001$). SEM studies revealed occurrence of cohesive type fractures in the ceramic body of samples belonging to Groups I & III, while there was adhesive failure at ceramo-metal junction of samples belonging to Group II. EDS Analysis supported the findings of SEM studies.

Conclusion: All the three ceramic systems fared better than the minimal recommended values stated by ISO 9693:1999 of 25MPa of which Orotig TiKrom was rated the best with values of 54.69 MPa. Vita TitanKeramik ranked second with values of 45.12MPa and the least values were obtained with Noritake Ti-22 with values of 27.76. The bond failure was predominantly cohesive in nature in case of Vita TitanKeramik and Orotig TiKrom; whereas adhesive failure was noticed in case of Noritake Ti-22.

Key Words: bond strength, bond failure, Titanium, ultra low fusing ceramics.

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Introduction

Noble metal alloys like Gold (Au), Platinum (Pt), Palladium (Pd) with the addition of Silver (Ag), Copper (Cu) and Zinc (Zn) have been used for cast metal restorations since the introduction of lost wax technique to dentistry around the turn of century by Taggart in 1906.[1] Noble metal alloys have been gradually replaced by base metal alloys due to the high cost and low sag resistance of the former.

In recent years, titanium has become a material of great interest in prosthetic dentistry. Titanium has been used in metal-porcelain restorations because of its several advantages,[2-5] like exceptional biocompatibility,[2-5,6] excellent corrosion resistance[2-5, 7] and high strength to low density (4.5 g/cm) ratio.[2-5] However, its limitations are high melting point and high chemical reactivity with oxygen, nitrogen, carbon and hydrogen at high temperatures.[8] Casting temperatures above 800°C produce an increasingly thicker oxidation layer, [9, 10, 11, 12]

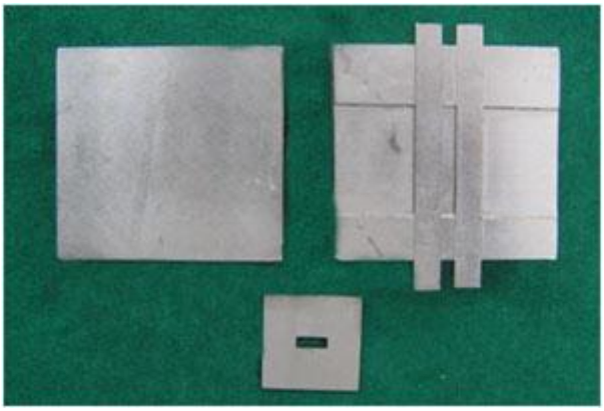

Fig. 1: Metal template for fabrication of resin patterns.

with proportionally weaker bond strength of ceramics to titanium. [9,10,11,12] Furthermore, the thermal expansion coefficient of the porcelain should be close to or slightly below that of titanium (9.6 X 1°-6°C). [13-15] Because of this, titanium requires low fusing porcelains for ceramometal restorations. Porcelains with firing temperatures around 750°C and adequate expansion coefficients are now available on the market as ultra low fusing ceramics.

To a great extent the success of the porcelain-fused-to-alloy restoration depends on the strong bonding between porcelain and the metal substructure.[16] Acceptable metal-ceramic restorations require metals and porcelains to be chemically, thermally, mechanically and esthetically compatible. With the increased popularity of Titanium as a ceramic metal more and more ultra low fusing ceramics are becoming commercially available.

Therefore this in-vitro investigation was designed to compare the flexural bond strengths of three commercially available ultra low fusing ceramic systems to Grade II Titanium and evaluate the type of bond failure through Scanning Electron Microscopy (SEM) and Energy Dispersion X Ray Spectrum (EDS)

Materials and Methods:

A Stainless Steel slab of dimensions 50mm X 45mm X 20mm was subjected to grinding by a Computerized Numerically Controlled (CNC) grinding machine (Anmol 200/400, Rajkot) to create a trough of dimensions 25.1 mm X 3.1mm X 0.6mm. Two adjacent slots of equal dimensions were created alongside for placement of Stainless Steel metal bars.

An auto-polymerizing acrylic resin (Figure 1, Pattern resin, GC Corp Tokyo, Japan) was flown into the slot and allowed to polymerize for 15 minutes under a pressure of 2 psi generated by a hydraulic bench press (KaVo, Germany). A total of 60 patterns were made. Defective patterns were rejected. Resin patterns were sprued with standardized short round sprues of 2.0 mm thickness (S-C Sprue wax, Schuler Company, Germany). Spruing of patterns to the rubber base was done.

Investing, de-waxing and burnout procedures were carried out as per the manufacturer’s instructions. Titanium casting for all the samples was done in a Titanium casting machine (OROTIG, Italy).

After casting, the samples were cleaned; any incomplete or defective castings were noted and these were rejected from the samples.

Finishing of samples was done with the help of Tungsten carbide burs, heatless discs, rotary aluminum oxide (100-800 grit) sand paper strips (Orotig, TITEC Italy) to ensure the complete removal of alpha phase. The verification of dimensions of the Titanium samples was done with the help of digital calipers (BAKER INDIA Ltd, Pune, least count 0.01mm) which was 25 x 3 x 0.5 mm +/- 0.01 mm, as required by DIN 13927.

A Dental X-ray unit (Confident dental company,

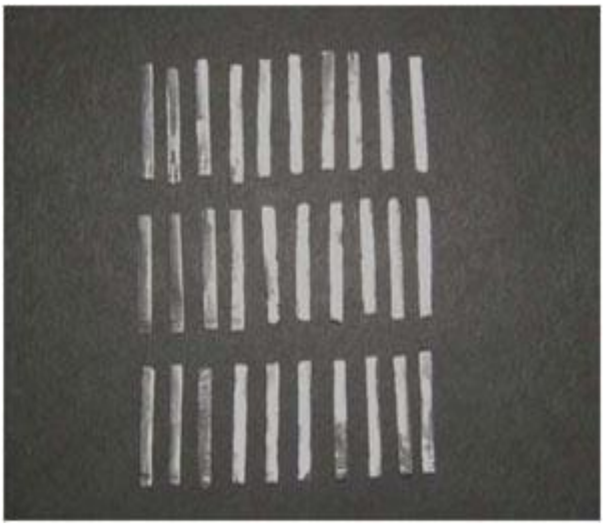

Fig. 2: Titanium specimen with ceramic build-up.

Bangalore) was used to obtain the radiographs on an occlusal radiographic film (Kodak corp. Japan). Samples with internal porosities were rejected.

From these selected samples 10 samples were allotted to each of three groups randomly. Each of the samples was given a number along with a mark denoting the group.

Ceramic build up of the samples (Figure 2) was done by using an alignment jig (dimension 8mmx3mmx1mm-DIN 13927) step by step using the manufacturers' instructions.

Flexural Bond strength tests for the samples were performed by using a 3-point bending test (Figure 3) on a Universal testing machine (No 5582, Instron Corp.

Fig. 3: Three-point bending test on a Universal testing machine.

Mass, U.S.A) (Figure 3) in compliance with Deutsches Institut für Normung (DIN) 13.927 draft. After the tests were completed, three samples, one from each group were selected randomly for the subsequent SEM /EDS studies.

Scanning Electronic Microscopy (SEM) (FEI- Quanta 200) was carried out to characterize the type and morphology of the fracture in representative specimens selected from each combination in which there was complete separation between porcelain and metal after the bending test. The area used was a 0.25 mm area in the centre of the Ti sample where porcelain was veneered. The magnifications at which the samples were photographed were 200 X, 800 X and 3000 X.

Table 1: Fracture Load and Flexural Strength for Different Groups.

Sample nos.	Group I		Group II		Group III	
	Load(N)	Flexural strength (MPa)	Load(N)	Flexural strength(MPa)	Load(N)	Flexural strength(MPa)
1	4.86	48.6	2.87	28.7	5.21	52.1
2	4.12	41.2	3.29	32.9	4.59	45.9
3	4.25	42.5	2.93	29.3	5.43	54.3
4	4.62	46.2	2.05	20.5	5.24	52.4
5	3.87	38.7	2.15	21.5	5.05	50.5
6	6.19	61.9	3.20	32.0	6.03	60.3
7	2.65	26.5	2.35	23.5	5.81	58.1
8	4.47	44.7	2.96	29.6	5.78	57.8
9	4.29	42.9	3.05	30.5	6.10	61.0
10	5.80	58.0	2.91	29.1	5.45	54.5
Mean	**4.51**	**45.12**	**2.76**	**27.76**	**5.46**	**54.69**

Table 2: Mean and Standard Deviations of Flexural Strength in Different Study Groups.		
Group	**Mean(MPa)**	**Standard Deviation**
I	45.12	9.86
II	27.76	4.35
III	54.69	4.70

Results

The maximum load to fracture the porcelain was recorded for each specimen (Table 1: Fracture load and flexural strength for different groups). All the means of the three groups [Group I – 45.12, Group II – 27.76, Group III – 54.69], were compared by one-way Analysis of Variance (ANOVA) and it was found that the mean bond strengths of the groups were not same. Group I & Group III had significantly higher bond strength values as compared with the Group II ($P< 0.001$). (Table 2: Mean and standard deviations of flexural strength in different study groups)

The comparison using the Student's unpaired "t" test to evaluate the inter Group Bond Strengths showed statistically significant difference in the bond strengths between Group I & Group III ($t = 2.76$ and $P< 0.05$), between Group I & II ($t = 5.09$ and $P< 0.001$) and between Group II & Group III ($t = 13.28$ and $P< 0.001$). (Table 3: Results of unpaired students' "t" test to compare the mean flexural strength in between two groups).

All the 10 (100%) samples of Group I & III gave bond strength values above the standard values of 25 MPa (ISO 9693:1999). In Group II, only 7 (70%) of the 10 samples were above the standard values of 25 MPa. However this difference is not statistically significant as the values of Fisher exact Test is $P = 0.1053$.

Scanning Electronic Microscopy

SEM of samples after fracture loading of the three groups revealed the presence of residual porcelain islands adhering to the metal surface of the substrate in

Table 3: Results of Unpaired Students' "t" Test to Compare the Mean Flexural Strength in Between two Groups.		
Source Of Variation	"t"	P- Value
Group I and Group II	5.09	<0.001 Highly significant
Group I and Group III	2.76	<0.05Statistically significant
Group II and Group III	13.28	<0.001 Highly significant

Table 4: EDS Analysis for Weight Percentages of Various Elements in Different Groups.						
	Group I (TitanKeramik)		**Group II(Ti-22)**		**Group III (TiKrom)**	
Element	Wt %	At %	Wt %	At %	Wt %	At %
O K	31.23	51.81	27.94	53.28	37.14	57.71
NaK	3.1	3.58			4.12	4.45
AlK	4.52	4.44	1.17	1.32	5.72	5.27
SiK	26.75	25.28	0.66	0.72	19.55	17.3
PtM	9.09	1.24				
K K	5.06	3.43			3.16	2.01
SnL	3.64	0.81			7.77	1.63
CaK	2.79	1.85			1.16	0.72
TiK	12.67	7.02	69.77	44.43	17.73	9.2
FeK	1.15	0.55	0.47	0.25	1.15	0.51
V K					1.23	0.6
CrK					1.05	0.5
MnK					0.22	0.1
Total	100	100	100	100	100	100

the Groups I & III, suggesting the occurrence of cohesive type fractures in the ceramic body - observations that attest to a better mechanical performance of these combinations. On the contrary there was no evidence of residual porcelain presence on the metal surface substrate in Group II. This meant a total loss of porcelain suggesting adhesive failure at ceramo-metal junction. This is further proved by the lower bond values.

EDS Analysis

The readings obtained for the EDS analysis are given in Table for EDS Analysis (Table 4: EDS Analysis for weight percentages of various elements in different groups). Since Ti is the major element of the metal used in the present study, its profile does not indicate that diffusion into the porcelain has occurred at the interface of every system.

Group I

Varying amounts of TiO2 present; Predominant Si in graph indicates there has been a cohesive failure of ceramic.

Group II

Predominant Ti and O2 and trace amount of Si seen which is indicative of ceramic area. The presence of O2 indicates that there is an intact oxide layer present over the surface and that the ceramic oxide has penetrated only to a minimal level. This graph confirms the fact that there has been a total debonding of the ceramic oxide layer from Titanium surface. This is indicative of total adhesive failure.

Group III

Graph shows the presence of O_2, indicative of oxide layer on the surface of Ti. The presence of Si is indicative of the fact that there is presence of ceramic layer, confirming a cohesive failure.

Discussion

Titanium forms an oxide coating over itself when cast. Its thickness can range from 5-10nm. This layer actually gives the corrosion resistance to the metal and makes it biocompatible. This oxide layer remains fairly stable up to 800°C. [2-5,9] The oxide layer over titanium increases in thickness over 800 ^{0}C resulting in weaker bond strength of ceramic to titanium. Therefore any ceramic that is fired over 800°C cannot be used. This prompted the development of ultra low fusing ceramics, which had firing temperatures of 800°C or less. [17, 18]

Togaya[10] investigated the compatibility of porcelain to cast pure titanium and suggested that acceptable bond strength was possible by reducing the difference of thermal expansion coefficient of the porcelain to approximately that of titanium. It is agreed that compatibility is said to exist when there is a mismatch in Coefficient of Thermal Expansion of about l X lO-6 /° C or less, so that porcelain remains under compression over the metal. The type of metal and the porcelain chosen for this study were compatible with each other in terms of their coefficient of thermal expansion.

There are various tests using different loading systems to evaluate the porcelain-metal flexural strength. The three point loading system first used by Wilson José Garbelini et al[19] has prevailed for brittle materials like porcelains and is contained in the project proposed by the German Standard Deutsches Institut für Normung (din 13.927). The American dental association (ADA) council also recommends it. This test was used for the present study.

The use of Titanium binder in Group I and the use of the base opaque in Group III (3 phase system) could probably be the reason for these groups exhibiting better mean flexural strengths than Group I. This may be due to an incorporation of various binding materials specific for titanium in them. The alloy composition of Grade II titanium is slightly different from the ASTM Grade II (Table I d). The lower bond values with this group could be the result of a thicker oxide layer. These readings of Group II were similar to what Zinelis et al[20] got, in contrast to values put forward by the manufacturer (>50MPa).

There may be various factors affecting bond strength between ultra low-fusing porcelains and titanium substrates. These include the thickness of the oxide layer formed on the metal surface,[19] diffusion of chemical elements during the firing of porcelain on the titanium producing variations in the oxide layer formed on the surface, differences in thermal expansion coefficients of the porcelains used. [4, 19] On the other hand, it is important to realize that the firing cycles used for porcelain build-up may worsen the fit of titanium copings to values that suggest no interferences to the detriment of clinical applications.[21]

The samples ruptured after the 3-point bending test and observed through E-SEM revealed a small amount of residual porcelain adhering to the Ti grade II in combinations with TiKrom and Titankeramik, but not with Ti-22. This finding was further strengthened by the EDS evaluation.

These results are similar to those found by Adachi, et al[22], Pang, et al[23], Yilmaz, Dincer[13] and Suasuwan, Swain[14]. Fracture involving predominant cohesive types was seen for combinations involving Ti grade II substrates and the Groups I and III. While in Group II, the fracture was of the adhesive type.

The bond of ceramic to titanium is a critical factor affecting the success of Titanium-ceramic restorations. The factors involved in the formation and modification of this layer should be observed and respected. Critical factors that influence the selection & use of a particular ceramic system for fabrication of Ti-ceramic restorations include: surface treatment applied to the substrate, the size of the particles used for sandblasting, adequate waiting time between sandblasting and ceramic-build up & the potential of certain chemical elements (bonding agents) when applied on titanium to enhance bond strength. [4, 14]

Conclusion

The bond strength of veneered ceramic to titanium is critical to the success of these prostheses. All the three ceramic systems fared better than the minimal recommended values stated by ISO 9693:1999 of 25MPa of which Orotig TiKrom was rated the best with values of 54.69 MPa. Vita TitanKeramik ranked second with values of 45.12MPa and the least values were obtained with Noritake Ti-22 with values of 27.76.

The SEM evaluation of a representative sample from each group was done to ascertain the type of bond failure occurring at the metal-ceramic interface. The bond failure was predominantly cohesive in nature in case of Vita TitanKeramik and Orotig TiKrom; whereas adhesive failure was noticed in case of Noritake Ti-22.

EDS evaluation confirmed the results of SEM study by identification of residual elements present at the metal-ceramic interface after de-bonding.

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